NASA CONTRACTOR REPORT

NASA CR-118

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AN INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

by A. Gilbert, B. C. Allen, and C. N. Reid

Prepared under Contract No. NASw-101 *by*
BATTELLE MEMORIAL INSTITUTE
Columbus, Ohio
for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • NOVEMBER 1964

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For sale by the Office of Technical Services, Department of Commerce,
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PREFACE

Work during the past year has progressed in three different directions. Accordingly, the experimental results are reported in three discrete sections, followed by a general discussion:

Section 1. Impact Testing of Chromium and Cr-35 At. % Re

Section 2. Tensile Testing of Hardness-Minimum Alloys:
Cr-0.5 At. % Ru, Cr-1.0 At. % Mn, and Cr-3 At. % Re

Section 3. Tensile Testing of Cr-17.5 At. % Fe and Cr-17.5 At. % Ru

Section 4. General Discussion.

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INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

by

A. Gilbert, B. C. Allen, and C. N. Reid

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SECTION 1. IMPACT TESTING OF CHROMIUM AND Cr-35 At. % Re

Cr Cr

INTRODUCTION

Although it has been known for almost 10 years that additions of rhenium to the Group VI-A refractory metals can under some conditions lead to enhanced ductility, the effect has not as yet been fully documented. In particular, the testing procedures used to investigate the ductilizing effect have been usually either tests of workability or slow-bend or slow-tension transition-temperature determinations. While such investigations have been and still are of great usefulness in interpreting the mechanism of the alloying effect, an evaluation of the structural potential of a particular alloy is more usually determined by a higher strain rate test, such as a determination of the impact transition temperature. Therefore, it was decided to determine the notch-impact transition temperature of chromium* and chromium-35 at. % rhenium to evaluate their relative mechanical properties at these higher strain rates.

EXPERIMENTAL PROCEDURE

Materials Preparation

Chromium rod, 1/4 inch in diameter, was prepared from iodide crystals compacted to form rectangular bars. Consolidation and fabrication consisted of consumable arc melting, extrusion 26:1 at 1250 C, and sheath swaging to an 80 per cent reduction in area at 900 C.

The Cr-35 at. % Re alloy was prepared by nonconsumable arc melting a mixture of iodide chromium and sintered rhenium of 99.99 purity, which was cast in cylinders. These alloy cylinders were then welded longitudinally to form a cylindrical electrode for consumable arc melting. The resultant billet was extruded 10:1 at 1700 C and then sheathed in evacuated molybdenum cans and swaged to an 80 per cent reduction in area at 1300 C.

* The work on chromium has been partially described in a previous NASA report (Twelfth and Thirteenth Quarterly Progress Reports, dated October 31, 1963), but a review will be included again for the sake of comparison with the present work.

[from cylinders 1 1/8 inch long by 0.225 inch in diameter, with a 0.040-inch-deep 45-degree notch around the middle. The notch radius was 0.005 inch.]

Testing Procedure

The fabricated material was in the form of 1/4-inch rod, which put an upper limit on the size of impact specimens which could be adopted. Reports in the literature(1,2)*, however, have shown that micro-impact specimens of the dimensions shown in Figure 1 have transition characteristics closely similar to those of standard V-notch specimens. Specimens of chromium and chromium-35 at. % rhenium were therefore ground, in the wrought condition, according to Figure 1. In the case of chromium, specimens were also ground to the same specification from recrystallized material to test the effect of specimen preparation. Of this latter group, some were re-annealed after the machining of the notch to remove the effects of residual stress. Table 1 summarizes the types of specimen preparation for both the chromium and the chromium-35 at. % rhenium alloy and also gives the interstitial-impurity analyses for both materials. The chromium samples, wrapped in tantalum foil, were annealed in a double quartz capsule, the inner capsule being evacuated and the space between the inner and outer capsules containing 10-cm pressure of argon. The annealing temperature was 1100 C and the time 1 hour. The chromium-35 at. % rhenium samples were placed in a tantalum can and were annealed for 1 hour at 1400 C under a dynamic vacuum of 1 x 10^{-5} mm of Hg, in a tantalum-element furnace. All the specimens were furnace cooled.

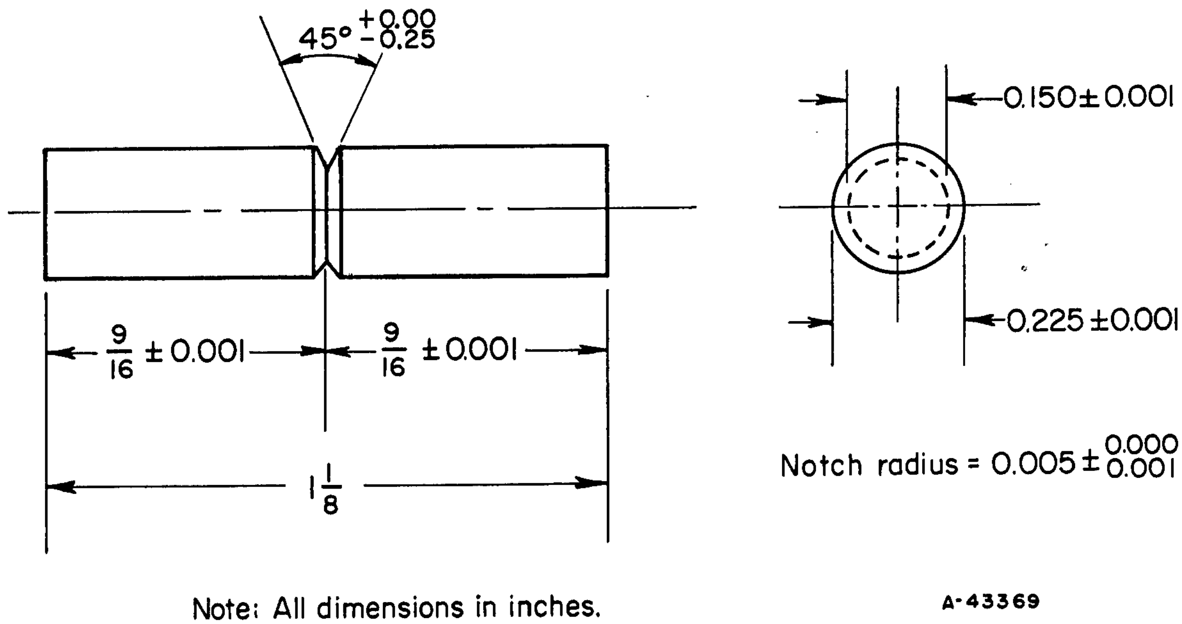


FIGURE 1. SPECIFICATIONS FOR MICRO-IMPACT SPECIMEN

The specimens were broken on a Tinius Olsen testing machine with a capacity of 16.7 ft-lb and an impact velocity of 11.4 ft/sec. The bottom half of the specimen was held in proper position by a steel adapter block, which allowed the pendulum to strike ~0.4 inch above the notch. A stainless steel cover block was made to fit fairly snugly over the protruding half of the specimen, and rest on the adapter block. This added to the thermal mass of the specimen assembly and could be removed immediately prior to impact. The adapter block, sample, and cover block were heated together in an air muffle furnace to slightly above the desired testing temperature, as indicated by a thermocouple embedded near the sample. After the temperature stabilized, the assembly was withdrawn from the furnace and clamped in the machine. Immediately after

*References are listed at end of report.

removal of the cover block, the specimen was broken. The specimen temperature at the time of fracture was taken from the time-temperature record starting with removal of the assembly from the furnace.

After testing, the broken samples were subjected to optical and electron-replica fractographic examination.

TABLE 1. DESCRIPTION OF MATERIALS AND SPECIMEN PREPARATION

Test Material	Lot No.	Method of Preparation	Impurity Content, ppm			
			C	O	N	H
Cr	1	Machined from wrought material, tested	15	20	2 ± 5	<. 3
Cr	2	Machined from annealed material (1 hr, 1100 C), tested	15	20	2 ± 5	<. 3
Cr	3	Machined from annealed material (1 hr, 1100 C), re-annealed (1 hr, 1100 C), tested	15	38	2 ± 1	<. 3
Cr-35 at.% Re	1	Machined from wrought material, tested	~30	137	13	<. 3
Cr-35 at.% Re	2	Machined from wrought material, annealed (1 hr, 1400 C), tested	~30	122	9	<. 3

EXPERIMENTAL RESULTS

Variation of Absorbed Energy with Temperature

The impact properties of chromium and chromium-35 at.% rhenium are presented in Figure 2 in the form of variation of absorbed energy with test temperature, and are tabulated in Table A-1 of the Appendix. The points in Figure 2 having an arrow above them refer to ductile specimens which bent and did not break. Such points are assumed to be equivalent to others on the upper plateau of absorbed energy, but the actual level of this plateau may have no quantitative significance.

As would be expected, the notch-impact transition temperature of chromium* was lower in the wrought (240 C) than in the recrystallized (~315 C) condition, the latter showing erratic behavior in the transition-temperature region. The grain size of this material lay in the range .06 to .18 mm, but no correlation between grain size and impact properties was observable. The highest transition temperature determined for chromium (380 C) was for specimens which had been re-annealed after machining.

The notch-impact transition temperature of chromium-35 at.% rhenium is not so well established as that of chromium because of the few specimens available, but the recrystallized alloy has a transition temperature close to 260 C, while the wrought seems to be brittle at higher temperatures.

*Taken to be the temperature at which the absorbed energy was half the maximum value.

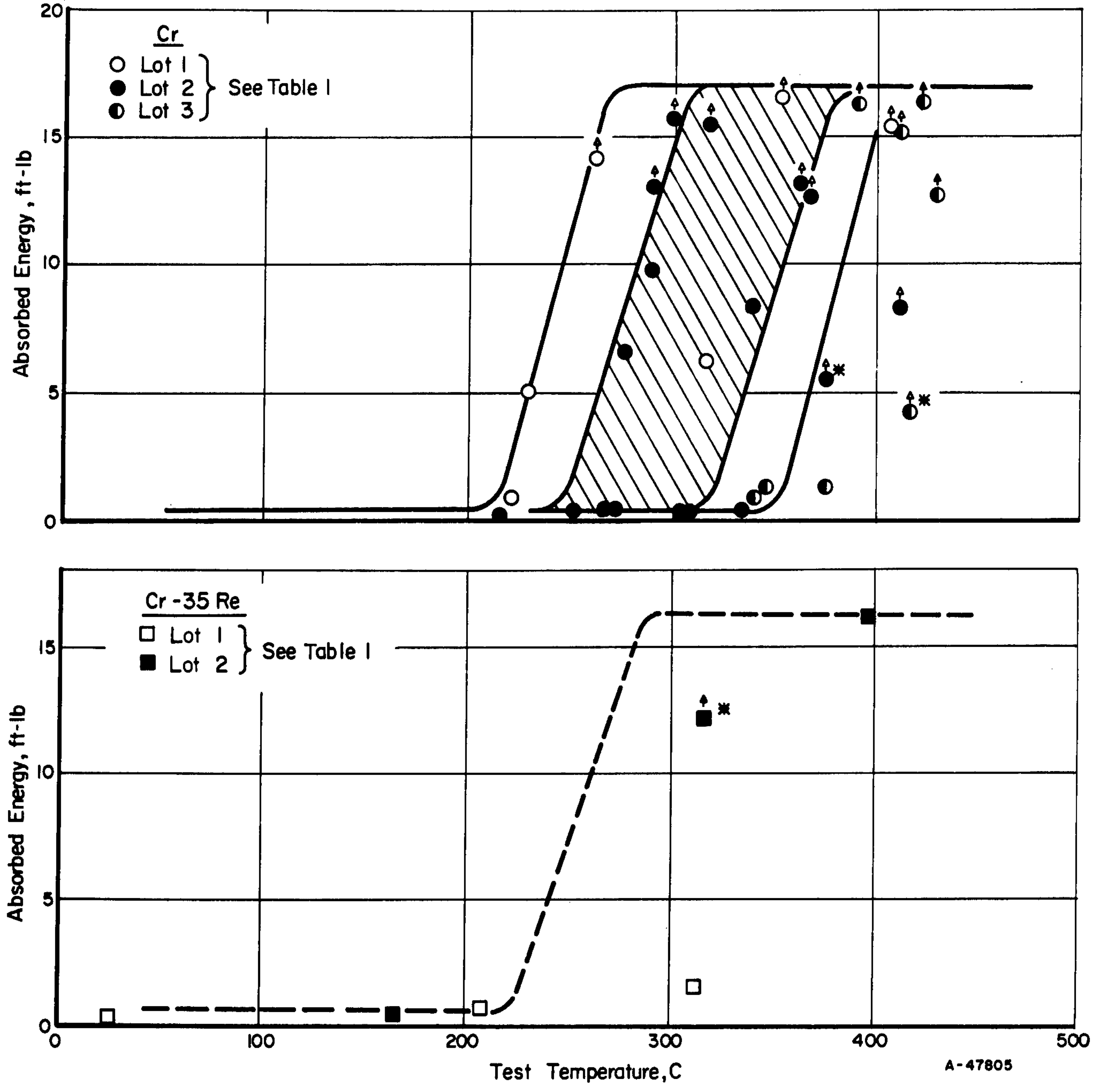


FIGURE 2. IMPACT BEHAVIOR OF NOTCHED SPECIMENS OF UNALLOYED CHROMIUM AND CHROMIUM-35 At.% RHENIUM

Asterisk (*) indicates specimen in which initial brittle fracture was arrested by ductile bending.

Fractography of Chromium

Brittle Specimens

Eleven specimens which broke into two pieces and absorbed less than 2 ft-lb of energy will be referred to as brittle. Fractographic examination however produced evidence of some plasticity in all of these specimens except the one tested at the lowest temperature, 216 C. This evidence was two-fold, and is illustrated in Figure 3.

FIGURE 3. TYPICAL FRACTURE SURFACE OF A BRITTLE SPECIMEN

Specimen P93.

A flattened facet was observed on the sides of the machined groove on the compression side of the specimen, where the upper half had been bent over to impact and plastically strain the lower half. Such an event can only occur as a result of plastic strain in the central, at that time unbroken portion of the reduced section. At the higher test temperatures, this evidence was augmented by observation of the ductile region on the fracture surface. In all cases where this facet was observed, a secondary origin of cleavage failure was observable at that part of the notch which was resubjected to a tensile stress as a result of the groove closing up on the compression side, making a secondary fulcrum at the extreme back edge of the specimen. The specimen broke by secondary cleavage failure propagating outwards from this origin, which in some cases involved backward propagation toward the ductile portion.

The mechanism by which the flattened facet is produced requires some plastic absorption of energy by material in the center of the original gage diameter, and it may reasonably be assumed that the size of the facet is a measure of the amount of plastic deformation occurring in this central portion.

For recrystallized brittle material, the size of the flattened facet increased slowly but monotonically with the test temperature, as did the value of absorbed energy. Grain-size scatter seemed to exert no noticeable effect. However one wrought specimen tested in this range, although brittle, absorbed approximately 5 times as much energy as the recrystallized specimens tested at similar temperatures. The size of the flattened facet, however, was similar to that for the recrystallized specimens tested in the same temperature region.

The fracture surfaces were generally typical of cleavage failure, and river lines could be traced back to a primary origin on the tension side of the specimen. For the brittle wrought specimen, and also for specimens machined after the anneal (Lots 1 and 2, Table 1), the fracture origin was never at the root of the notch but always between two and seven grain diameters in from the root. Grain boundaries were found to be the source of fracture, where an identification could be made. For specimens which were annealed after machining, however (Lot 3, Table 1), the primary fracture origin was observed to be at the notch root.

A change in the character of some of the cleavage facets could be detected at the higher test temperatures. An example of this change is shown in Figure 4 which contrasts typical river lines in a specimen tested at 266 C (Figure 4a) with those in a specimen tested at 375 C (Figure 4b), the highest temperature at which any specimen proved brittle. In the former specimen, the river lines are fairly sharp and well-defined, whereas at the higher temperature the lines are much less straight and are difficult to distinguish from lines which are thought to be evidence of plastic flow.

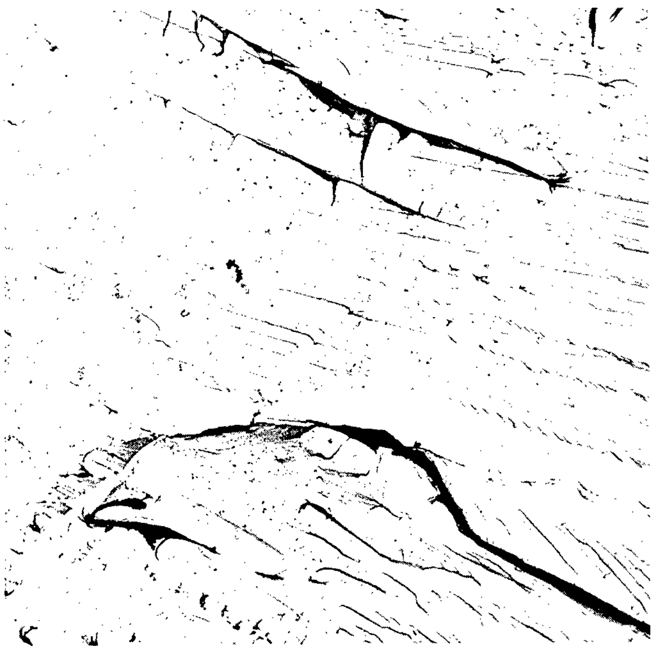
A metallographic cross-section through one-half of the sample proving brittle at the high temperature showed many instances of microcracks, examples of which are shown in Figure 5. Progressive sectioning showed that these cracks were not side branches of the main cleavage crack but were isolated examples of cracks which had initiated but had not propagated.

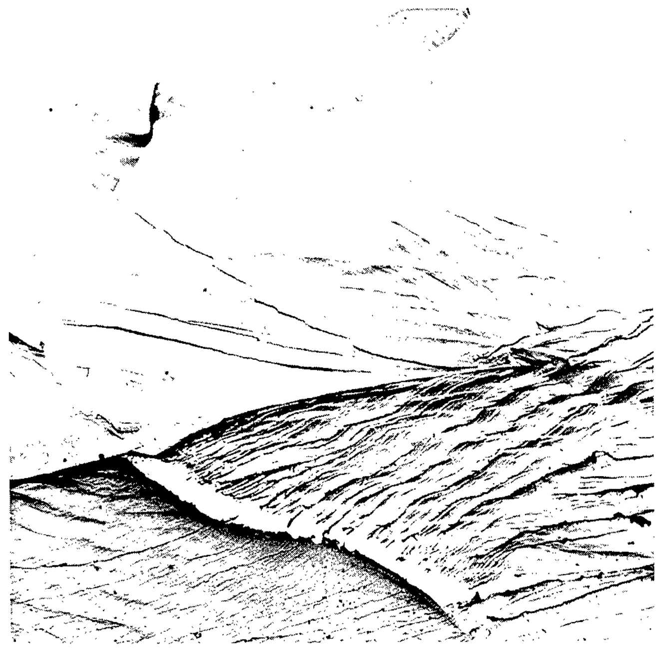
Semiductile Specimens

Five specimens which broke into two pieces after absorbing between 5 and 10 ft-lb of energy will be treated as semiductile specimens. These specimens, tested in the region of the transition temperature, deformed initially by plastic straining (during which time the bulk of the energy was probably absorbed), but fractured by cleavage. The origin of this cleavage was found to be at or near the notch surface at that point which was first subjected to tension as a result of specimen bending, as already discussed.

Ductile Specimens

Fifteen specimens which did not break into two pieces are considered ductile. Generally, such specimens bent through about 70° and had no fracture surface as such. The material close to the notch was severely strained and had an appearance similar to that of a normal tensile specimen which had been strained close to necking. Two exceptions to this behavior were the specimens shown starred in Figure 2 at test temperatures of 371 C and 415 C. These specimens, although they did not break, showed initial cleavage fracture, in the tension sides of the specimens, which was arrested by ductile flow in the compression half of the specimen before complete failure could occur. The origins in these specimens were 10 to 20 grain diameters from the notch.





2500X E766D 2500X E693C

a. Tested at 266 C b. Tested at 375 C

FIGURE 4. COMPARISON OF CLEAVAGE FACETS REVEALED BY ELECTRON REPLICAS OF SPECIMENS TESTED AT 266 C (a) AND 375 C (b)

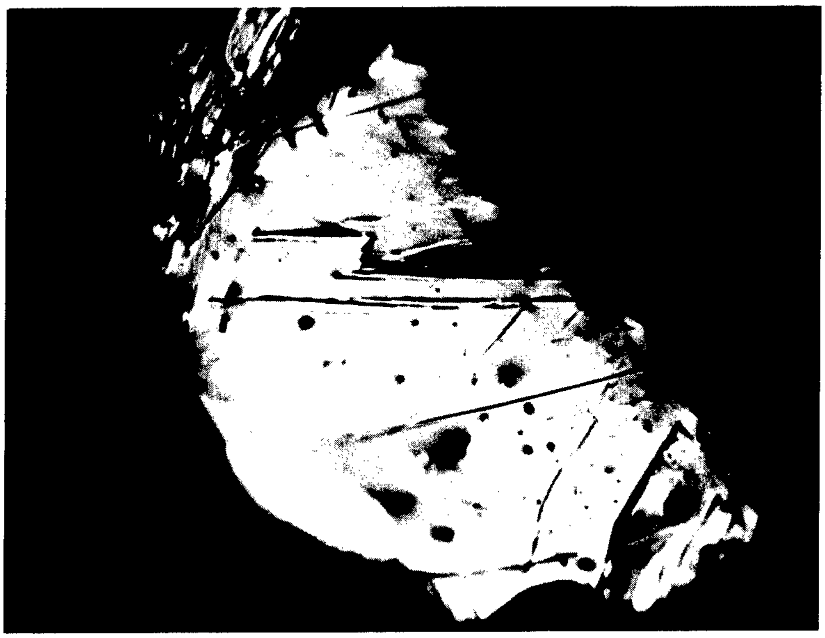
500X 12333

FIGURE 5. MICROCRACKS OBSERVED IN THE CHROMIUM SPECIMEN TESTED AT 375 C

Fractography of Cr-35 At. % Re

Brittle Samples

One recrystallized and three wrought samples failed in a brittle manner, but the appearance of the fracture surfaces was very different from that of brittle chromium samples. By eye, the surfaces had a dull over-all appearance, with occasional shiny facets. Under the optical microscope, the dull areas could be resolved as mainly grain boundaries similar to that shown in Figure 6, an example taken from the recrystallized

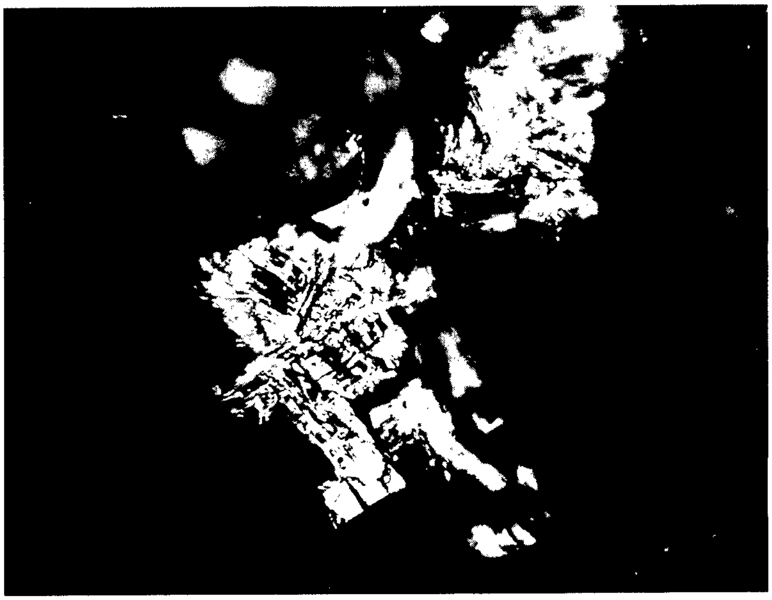


250X 9176

FIGURE 6. EXPOSED GRAIN BOUNDARY ON THE FRACTURE SURFACE OF THE BRITTLE RECRYSTALLIZED SAMPLE OF Cr-35 At. % Re

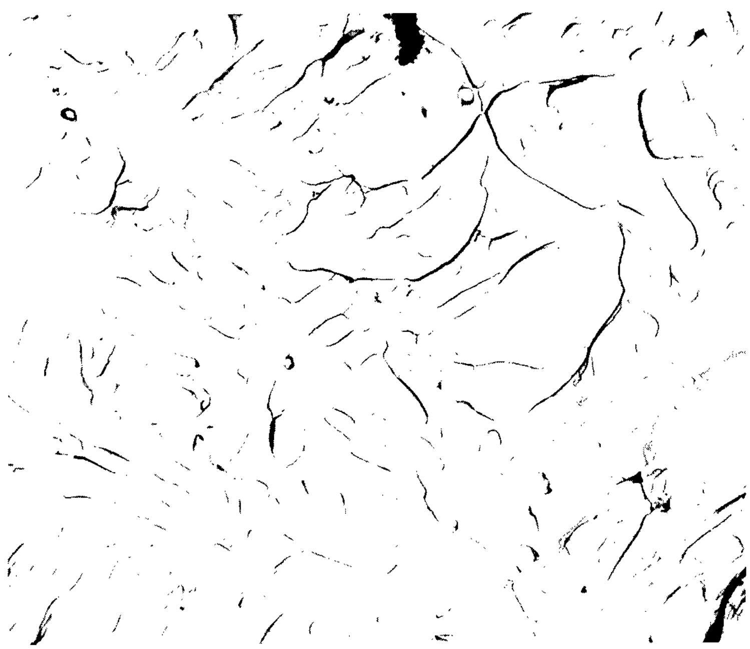
specimen. Precipitates and twins are visible at the exposed boundary. The shiny areas were resolved to be either cleavage facets with normal river lines, or, more commonly, facets similar to those shown in Figure 7a, on which it was very difficult to make out the details. Negative electron replica fractographs, similar to that in Figure 7b, showed these facets to be free of normal river lines. The markings on the surface seem to be similar in nature to river lines, but with a more geometric arrangement, and do not have the over-all unidirectional nature usually connected with river lines. This type of facet will be referred to as rhenium-modified cleavage. Stereographic replica work gave the indication that such surfaces are cleavages, apparently alternating between two different levels.

In addition to grain-boundary area and cleavage failure, the specimen tested at the highest temperature (316 C) had signs of appreciable plastic strain in the form of "ductile dimples". The grain-boundary areas, cleavage areas, and ductile areas were found at random across the fracture surface, frequently occurring close together.



330X 10680

a. Optical Fractograph



3,375X E905B

b. Negative Electron Replica Fractograph of Facet Similar to That Above

FIGURE 7. RHENIUM-MODIFIED CLEAVAGE FACETS IN A BRITTLE WROUGHT SPECIMEN OF Cr-35Re

Examples of this are shown in Figure 8a, a cleavage area next to a grain boundary, and in 8b, which shows a ductile region next to a grain boundary. This latter grain boundary has wavy lines across its surface, which are thought to represent prefracture slip within the grains. This same sample was the only one which, under the optical microscope, showed signs of ductility in the form of a small plastically strained region at the front of the notch and a flattened facet at the rear.

The grain boundary area and cleavage area were about equal to each other in the fracture surfaces and did not vary greatly over the range room temperature to 316 C. The major fractographic difference lay in the appearance of ductile dimples at the highest temperature.

The fracture surface of the brittle, recrystallized sample had, in addition, one other type of facet shown in Figure 9. Such facets seemed completely flat with no evidence of precipitates, but usually bore some pattern of a sharply angular nature.

Figure 9a shows the facet photographed optically at 330X, while 9b and 9c are negative replica electron fractographs at much higher magnifications, showing the detail in areas A and B, respectively. Measurements of the angles between the twin intersections on surface B, shown in 9c, are consistent with the surface having Miller indices (112) or (111) but not (100) or (110). This implies that such surfaces are actually twin/matrix interfaces rather than (100) cleavage planes. There are insufficient twins on surface A to permit such a test. Stereo pairs at about 5000X showed the "fingers" obvious in 9b were in fact extensions of the surface at the upper right, which was parallel to but above the surface at lower left.

This specimen showed signs of ductility in the form of a flattened facet.

Semiductile Samples

On the basis of the definitions used in describing the chromium fractography, the recrystallized sample tested at 395 C fell into the semiductile category. In addition, the specimen tested at 318 C was so close to breaking into two pieces that a complete break could be made by hand at room temperature, and so both specimens will be described in this category.

Both these specimens started to strain plastically and then failed in a brittle manner on the second subjection to tension. For the specimen tested at the lower temperature, this brittle failure apparently occurred as a result of a flaw introduced during fabrication which was deep enough to persist as a slight surface flaw after the specimen had been machined.

The specimen tested at the highest temperature, shown in Figure 10, had wavy markings across the exposed grain boundaries. As in the case of chromium, these markings are thought to represent slip taking place within the grain.

1870X E913A

a. Grain Boundary Area Above Rhenium-Modified Cleavage Area

13,725X E904D

b. Ductile Dimples Next to Grain Boundaries Showing Many Lines

FIGURE 8. NEGATIVE ELECTRON REPLICAS SHOWING EXAMPLES OF DIFFERENT TYPES OF FRACTURE SURFACE IN A BRITTLE WROUGHT Cr-35 At.% Re SAMPLE

330X 11116

a. Optical Fractograph

3960X E911A

b. Electron Replica in Area A, above

2750X E911D

c. Electron Replica in Area B, above

FIGURE 9. FLAT FACET FREQUENTLY OCCURRING IN BRITTLE RECRYSTALLIZED SAMPLE

750X 10345

FIGURE 10. EXPOSED GRAIN BOUNDARY ON THE SURFACE OF THE Cr-35 At. % Re SPECIMEN TESTED AT 395 C

DISCUSSION

The ductile-brittle transition temperature of chromium under the severe conditions of notch-impact testing was much higher than under the less rigorous conditions of slow tension. Recrystallized chromium similar in composition and method of preparation to that used in these tests proved ductile at or about room temperature at a strain rate of approximately 10^{-4}/sec, whereas, depending on history, the transition temperature under notch impact lies between 240 and 380 C at a strain rate of 10^2-10^3/sec. Although the present chromium is significantly purer than any tested previously, these temperatures are close to earlier determinations of the impact transition temperature of chromium summarized in Table 2. It has been suggested previously(7) that low tensile transition temperatures in chromium are achieved by making the initiation stage more difficult, rather than by changing the propagation characteristics. The agreement between the present work and earlier impact work lends support to this hypothesis, since it is apparent that cleavage failure can propagate quite as readily in this as in earlier, less pure chromium, despite its low tensile transition temperature.(7,8)

It seems likely that the different transition temperature of Lot 2 material (~310 C) from Lot 3 (~380 C) reflects the initiation aspect of failure rather than propagation. It is probable that machining the notch in annealed material (Lot 2) produced some plastic strain at the notch root; fracture could initiate only in the interior of the specimen, which allowed some energy absorption close to the root. Re-annealing such specimens (Lot 3) apparently removed the worked structure near the notch, permitting fracture to be initiated close to the surface under conditions where the tensile stresses are highest.

TABLE 2. COMPARISON OF TENSILE AND IMPACT TRANSITION TEMPERATURES OF CHROMIUM

Preparation History	Type of Test	Interstitial Content, ppm C	O	N	Transition Temperature, C	Strain Rate, in./in./min.	Reference
Wrought (80% at 900 C)	Notched micro Izod	15	20	2±5	+240	$\sim 10^4$	This work
Wrought, recrystallized grain size 0.08-0.10 mm	Notched micro Izod	15	20	2±5	+380	$\sim 10^4$	This work
Arc cast	Unnotched Charpy	<50	<300	20	<400	$\sim 10^4$	3
Cold pressed, sintered	Unnotched Charpy	<100	150	200	~450	$\sim 10^4$	4
As-drawn wire (75% at 350-100 C)	Miniature unnotched Charpy	<50	40	20	+110	$\sim 10^4$	5
As-drawn and recrystallized wire	Miniature unnotched Charpy	<50	40	20	~120	$\sim 10^4$	5
Electrolytic Cr+ 0.5Ti+6MgO, hot extruded	Unnotched Izod	--	--	--	~250	$\sim 10^4$	6

The still lower transition temperature of the wrought material (Lot 1), however, probably reflects a change in both initiation and propagation. The change in propagation characteristics is best shown by the wrought specimen failing at 220 C. Although brittle, this specimen exhibited the same amount of plastic flow on the fracture surfaces as recrystallized samples at this temperature yet absorbed five times more energy. This increase probably reflects a greater energy per cleavage facet involved in crack propagation through a worked structure.

The wavy nature of the cleavage surface shown in Figure 4b is interpreted as gross plastic flow taking place ahead of the propagating crack, which is almost sufficient to blunt and arrest the crack. This evidence taken together with the observation of microcracks in the same specimen suggests that notch impact testing comes close to determining the propagation characteristics of a crack in chromium, while it is believed that the tensile transition temperature reflects the initiation process.[7]

The impact properties of the Cr-35Re alloy proved similar to those of chromium. This similarity in terms of temperature, however, ~260 for recrystallized material, is not paralleled in terms of mechanisms. The alloy fractography showed evidence of

ductile failure, grain-boundary failure, and what is thought to be cleavage failure, often occurring in adjoining grains of a specimen. The small number of specimens available and therefore the restricted range of testing conditions employed make interpretation of this fracture behavior particularly difficult. However, it is apparent that the worked material has, if anything, a higher impact transition temperature than the recrystallized, which directs attention to the role of twinning as a deformation mode, the recrystallized material twinning much more readily than the wrought.

The most likely explanation for this unusual fracture behavior seems to be in the readiness of the grain boundaries to part. Some ductile dimples were observed in even the brittle specimens, which implies that if the grain boundaries could hold together, the intragranular material would be able to deform in a ductile manner. When grain boundaries do part in such large numbers, however, cleavage is initiated in those grains which are not suitably oriented for grain-boundary parting, due to the fact that such grains are "impacted" by the running grain-boundary crack. Since this cleavage may be initiated at more than one edge of the grain, cleavage propagates from more than one source in more than one direction. This may then account for the more geometrical appearance of the rhenium-modified cleavage. This readiness of the grain boundaries to part however is only relative. Tension tests at 10^{-4}/sec have shown[8] that similar failures, largely grain boundary, occur at liquid-nitrogen temperature at a stress of about 170,000 psi but not in a test at room temperature where the maximum engineering tensile stress before ductile failure is approximately 130,000 psi. This implies, therefore, that a stress of the order of 150,000 psi is necessary to cause grain-boundary parting. A linear extrapolation of the strain-rate sensitivity of Cr-35 at.% Re in the range 200-300 C shows that the yield stress in this range, ~70,000 psi, should increase by only about 10 per cent for an increase in strain rate of 10^6 times. This should not even reach the twinning stress in this temperature range (~90,000 psi) and certainly not the fracture stress. It appears therefore that the strain-rate sensitivity must increase considerably at the higher strain rates in order to explain the anomaly.

CONCLUSIONS

(1) The notch-impact transition temperature of iodide chromium is 240 C in the wrought and up to 380 C for the recrystallized conditions.

(2) Negative electron replicas of specimens failing by cleavage just below the transition temperature are interpreted as showing pronounced slip during failure.

(3) The observation of microcracks in a specimen fractured in a brittle manner at 375 C suggests that the notch-impact test reflects the propagation transition temperature for chromium.

Cr

(4) The ductile-brittle transition temperature of recrystallized Cr-35 at.% Re under notch-impact conditions is 260 C, and it is somewhat higher for wrought material. The difference is believed to be due to the greater ease of twinning in the recrystallized material.

17

(5) Cr-35 at. % Re fractures by grain-boundary failure, ductile failure, or cleavage. The unusual nature of the cleavage facets is thought to be due to cleavage propagating from more than one direction, or on other occasions, that twin/matrix interfaces form the fracture surface.

SECTION 2. TENSILE TESTING OF THE HARDNESS-MINIMUM ALLOYS, Cr-0.5 At.% Ru, Cr-1.0 At.% Mn, and Cr-3 At.% Re

Cr

INTRODUCTION

In a previous phase of this program[9,10] a screening study was made to determine the effect of dilute alloying on the mechanical properties of chromium. In this earlier study, room-temperature hardness was determined as a function of the concentration of different alloying additions. Generally, a minimum in hardness occurred at approximately 1 at.% alloying addition, and it was hoped that this minimum in hardness was accompanied by a corresponding increase in ductility. The present section describes a program designed to see whether such a ductilizing effect existed in selected hardness-minimum alloys tested in tension.

EXPERIMENTAL

Compositions Tested

The alloys chosen for tensile testing were Cr-0.5 at.% Ru, Cr-1 at.% Mn, and Cr-3 at.% Re. The first of these alloys was tested on the basis that in the earlier hardness-measurement program[9,10], the ruthenium series of alloys exhibited a more pronounced minimum than any other series. Compared with a hardness in the unalloyed chromium of 130 VHN, Cr-0.5 at.% Ru had a hardness of 110 VHN. It was therefore felt that this considerable decrease in hardness made the Cr-0.5 at.% Ru alloy a good one on which to seek the correlation between reduced hardness and increased ductility. The Cr-1.0 at.% Mn was chosen as an analog to the Cr-1.0 at.% Re alloy tested in a previous phase, Re and Mn being in the same group of the periodic table. Finally, the Cr-3 at.% Re was prepared as the chromium equivalent of a W-3 at.% Re alloy which has shown considerable promise in other NASA testing. In addition it complements the Cr-1 at.% Re alloy already tested, in terms of investigating the rhenium alloying effect at lower, more economically feasible alloying levels.

Rods of all three compositions were prepared by a combination of nonconsumable arc melting, hot gas-pressure bonding, rod rolling, and swaging. Arc-melted buttons were crushed, placed in evacuated stainless steel cans, and consolidated by 10,000-psi helium for 3 hours at 1340 C. The composites were rod rolled to 55 per cent reduction in area at 1200-1300 C and swaged to 70 per cent reduction in area at 1200 C to 1/4-inch-diameter rod.

Chronologically, the ruthenium and rhenium alloys were available in rod form before the manganese alloy, and annealing studies were made on these two alloys to determine a suitable recrystallization treatment. Small lengths were cut from rods of Cr-0.5 at.% Ru and Cr-3 at.% Re and annealed for 1/2 hour at temperatures between 1000 and 1400 C. Hardness determinations on the annealed specimen, summarized below in Figure 11, showed that a 1/2 hour anneal at 1300 C would recrystallize both alloys.

Although no annealing studies were made on the Cr-1 at.% Mn alloy, it was assumed that its behavior would be similar and that a 1300 C anneal would lead to complete recrystallization.

A summary of the interstitial analyses for these materials in the annealed condition is presented in Table 3.

TABLE 3. INTERSTITIAL ANALYSES OF HARDNESS-MINIMUM ALLOYS

Alloy Composition, at. %		Interstitial Content, ppm				
Nominal	Analysis	C	O	H	N	
Cr-0.5Ru	0.55Ru	~30	56(a)	0.4(a)	17(a)	50(b)
Cr-3Re	1.8Re	~30	278(a)	<0.6(a)	17(a)	50(b)
Cr-1Mn	2.0Mn	~30	156(a)	<0.3(a)	36(a)	50(b)

Note: The carbon content was estimated from previous analyses, which have always been in the range 15-50 ppm.

(a) Vacuum fusion.
(b) Micro-Kjeldahl.

Tensile Testing

Specimens were ground from the as-received rod to the shape shown in Figure 12, and electrolytically polished in 9:1 perchloric/acetic acid solution to remove approximately 5 mils from a gage diameter. Some samples were then annealed for 1 hour at 1300 C in a dynamic vacuum of 1 x 10^{-5} mm. Before testing, another 5 mils was removed from a diameter by further electropolishing.

Tensile testing was carried out in air at a strain rate of 7 x 10^{-5}/sec on an Instron testing machine equipped with a vertical wire-wound tube furnace. Tests were performed at temperatures between ambient and 700 C, and the ductile-brittle transition temperature was determined for all three alloys in the as-worked and the annealed condition. Where sufficient material was available, the transition temperature was also determined for material in the wrought stress-relieved condition, the stress relief consisting of a 1-hour vacuum anneal at 870 C.

Figures 13, 14, and 15 show the grain structure of the 3 alloys in the as received and also in the annealed condition.

EXPERIMENTAL RESULTS

The tensile properties of three dilute chromium-base alloys are presented in Figures 16, 17, and 18, and are tabulated in Table A-2 in the Appendix. All three alloys yielded discontinuously in the annealed condition, exhibiting stress-strain curves similar

23

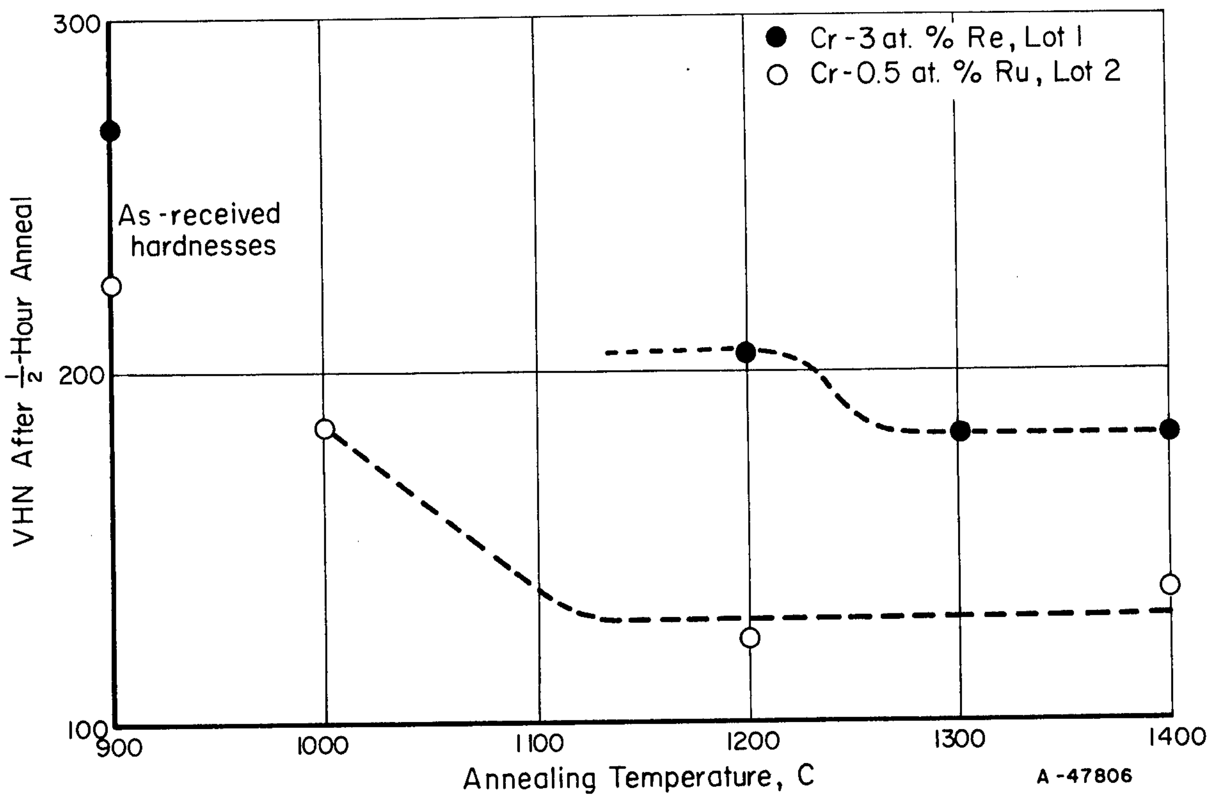


FIGURE 11. EFFECT OF ANNEALING TEMPERATURE ON HARDNESS OF Cr-0.5 At.% Ru AND Cr-3 At.% Re ALLOYS

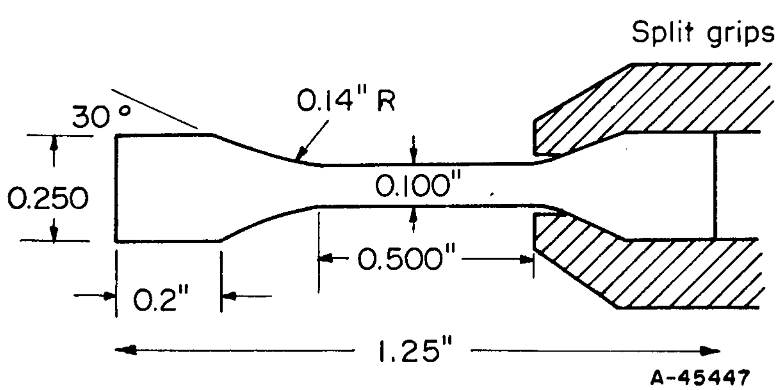
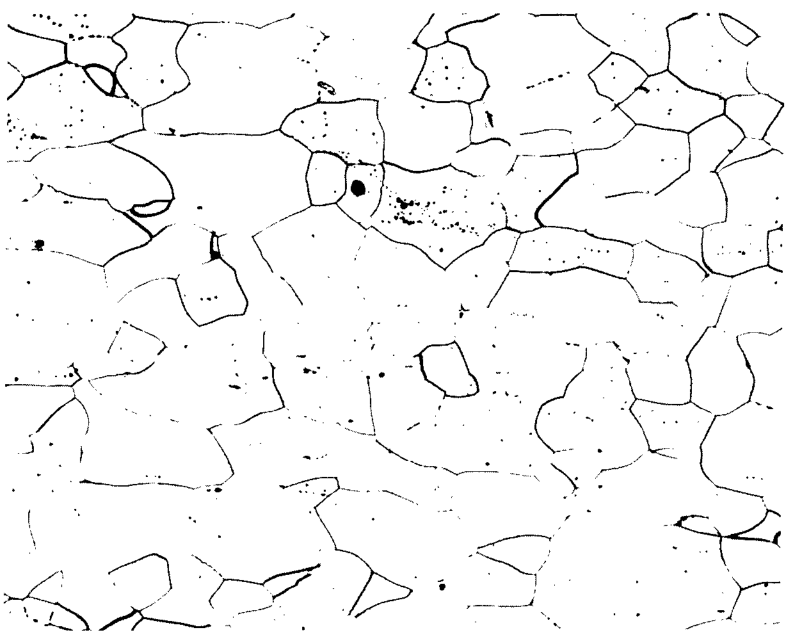


FIGURE 12. SPECIFICATIONS OF TENSILE SAMPLE

100X 9180

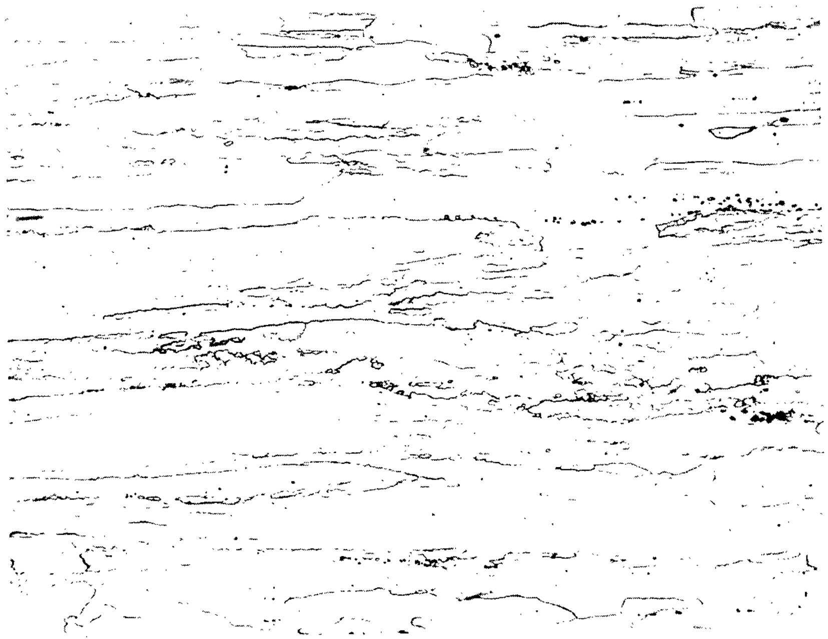
a. Lot 1, As Received



100X 9179

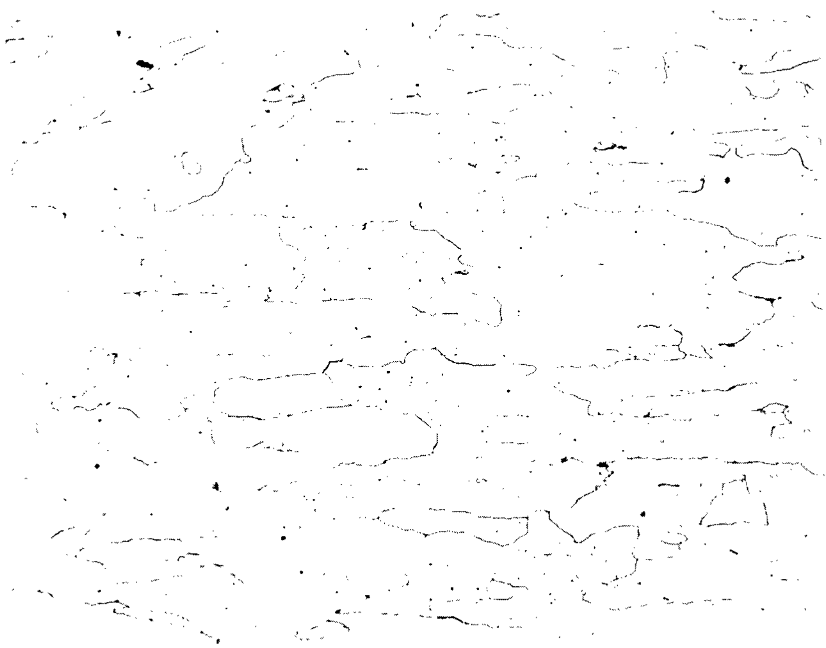
b. Lot 2, Annealed 1 Hour at 1300 C

FIGURE 13. GRAIN STRUCTURE OF Cr-0.5 At. % Ru



100X 9940

a. As Received



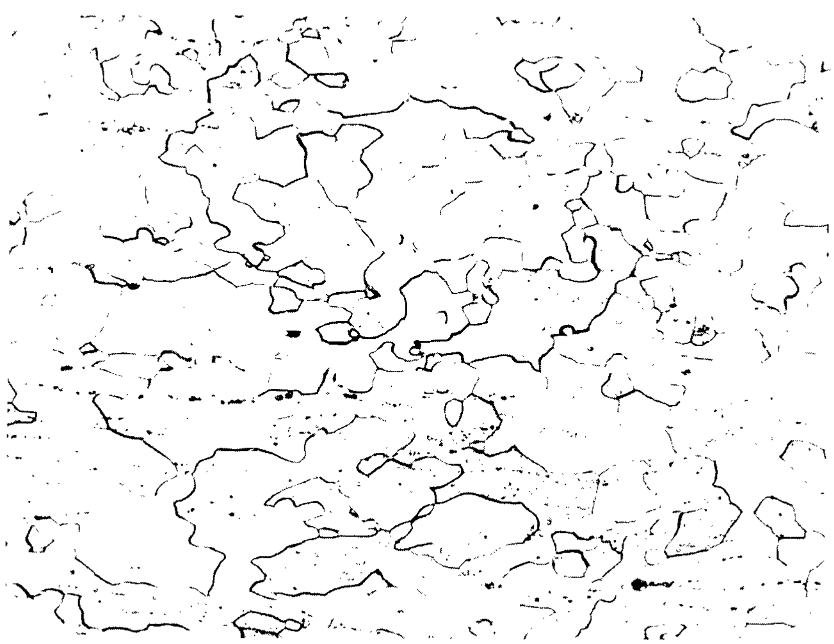
100X 9938

b. Annealed 1 Hour at 1300 C

FIGURE 14. GRAIN STRUCTURE OF Cr-1Mn

100X 9942

a. Lot 1, As Received



100X 9941

b. Lot 2, Annealed 1 Hour at 1300 C

FIGURE 15. GRAIN STRUCTURE OF Cr-3Re

to those obtained with chromium but at a stress level reflecting the effect of solid-solution strengthening; that is, the yield stresses increasing progressively with the nominal alloying composition.

The approximate ductile-brittle transition temperatures are presented in Table 4.

TABLE 4. TRANSITION TEMPERATURES OF HARDNESS-MINIMUM ALLOYS

Cr

Nominal Alloy Composition, at.%	Structural Condition	Transition Temperature, C
Cr-0.5Ru	As received	~205
Cr. 0.5Ru	Annealed 1300 C	~205
Cr-1Mn	As received	~230
Cr-1Mn	Stress relieved 875 C	>230
Cr-1Mn	Annealed 1300 C	~85
Cr-3Re	As received	~370
Cr-3Re	Stress relieved 875 C	~220
Cr-3Re	Annealed 1300 C	~450

> 8

One qualitative difference worthy of mention is the behavior of these materials in the strain-aging region, 300 to 500 C. The stress-strain curves showed two different types of serrations regular and irregular, examples of which are presented in Figure 19. Table 5 summarizes and describes the type of serrations which were observed. Since this was only a screening program, an insufficient number of tests were made to investigate the effect fully, but in general it seems that the regular serrations are associated with the higher alloy compositions and occur at higher temperatures than do the irregular serrations.

TABLE 5. TYPES OF SERRATIONS OBSERVED IN STRESS-STRAIN CURVES OF HARDNESS-MINIMUM ALLOYS

Alloy	Specimen	Test Temp, C	Type of Serrations
Cr-0.5 at.% Ru	1-1	400	Irregular
	1-11	350	Irregular
	2-4	315	Irregular
	2-7	246	Irregular
Cr-1.0 at.% Mn	5	400	Both irregular and regular
Cr-3 at.% Re	1-2	500	Regular
	2-3	454	Regular
	2-7	347	Irregular

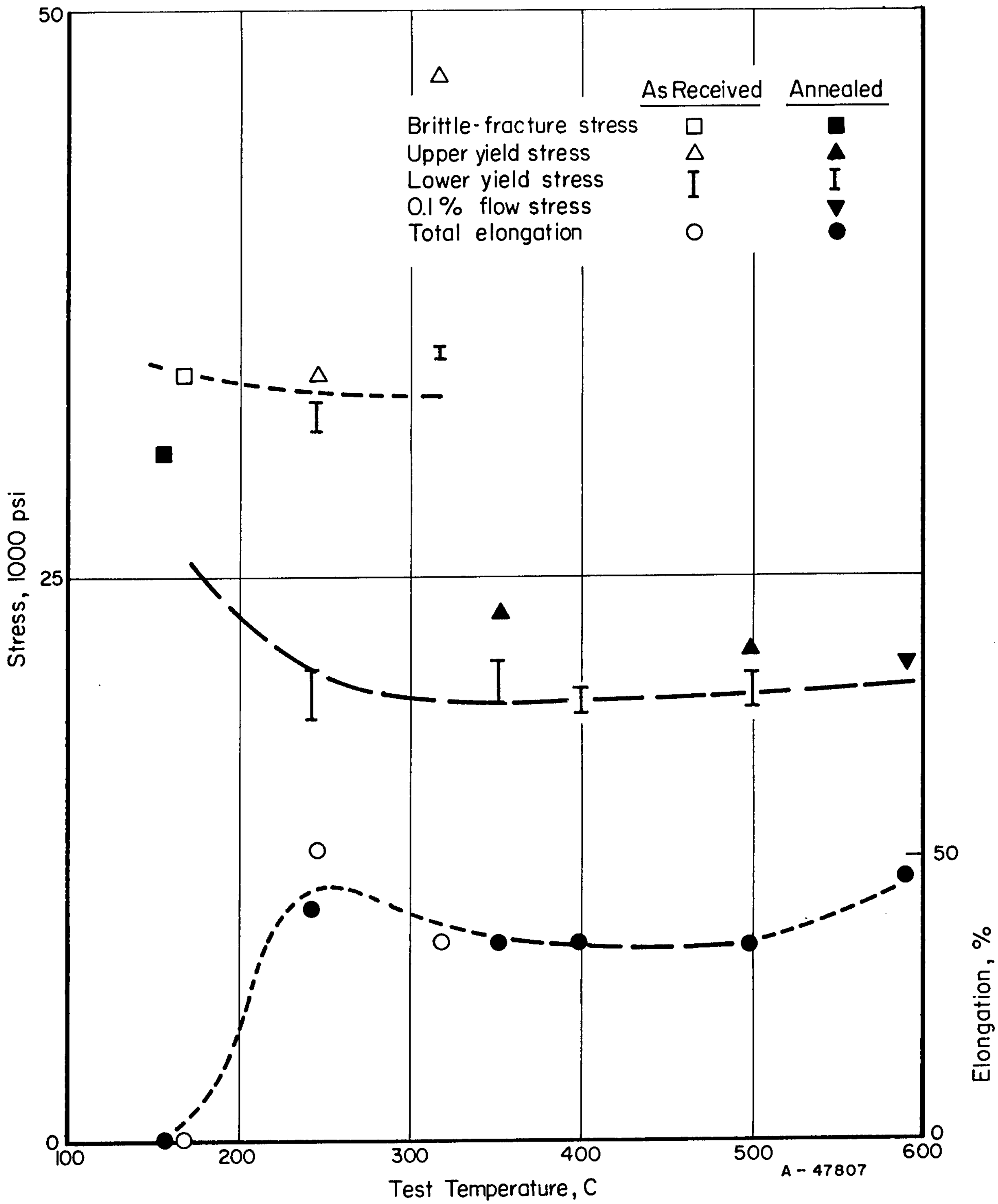


FIGURE 16. MECHANICAL PROPERTIES OF Cr-0.5 At.% Ru AS A FUNCTION OF TEMPERATURE

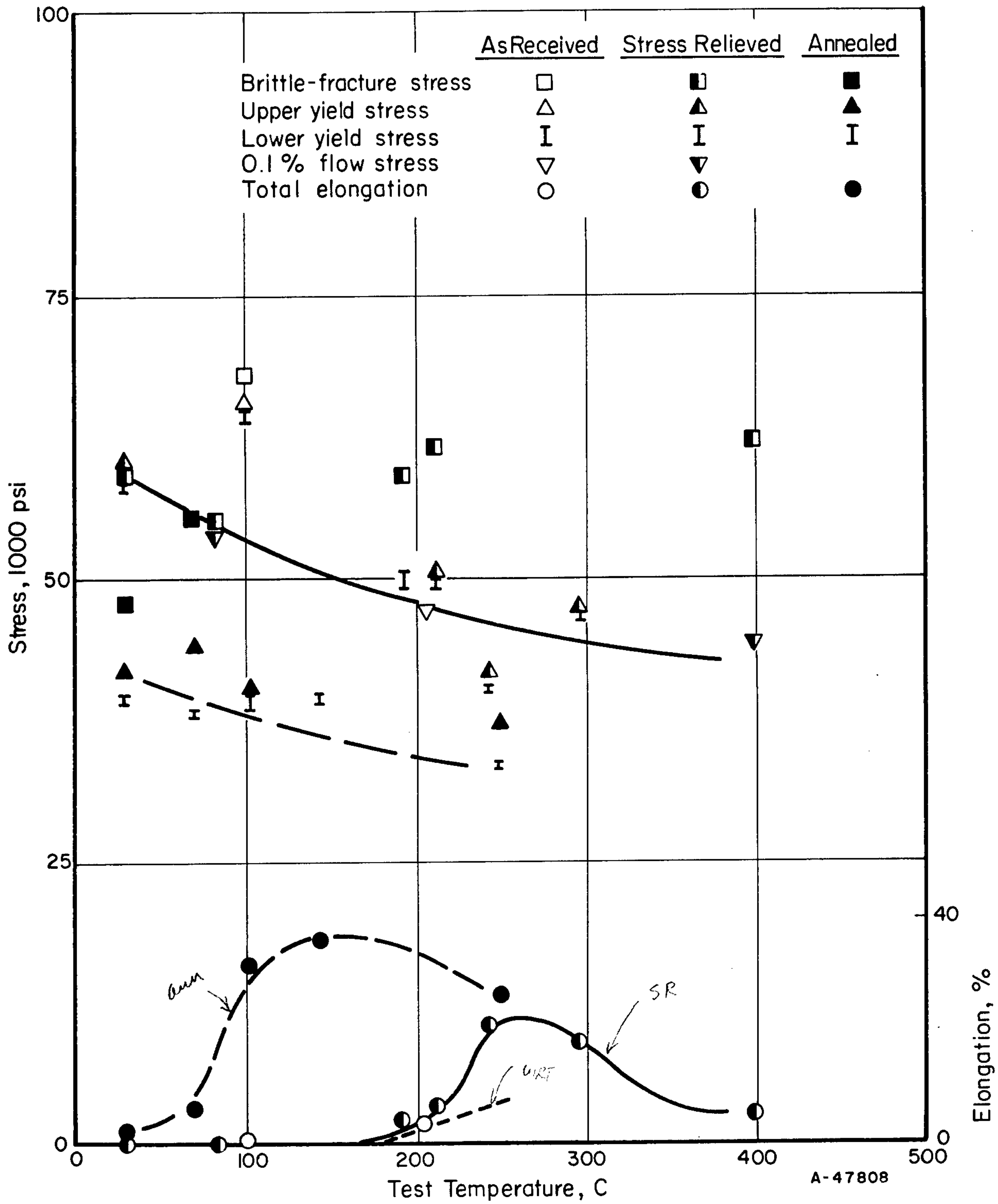


FIGURE 17. MECHANICAL PROPERTIES OF Cr-1 At. % Mn AS A FUNCTION OF TEMPERATURE

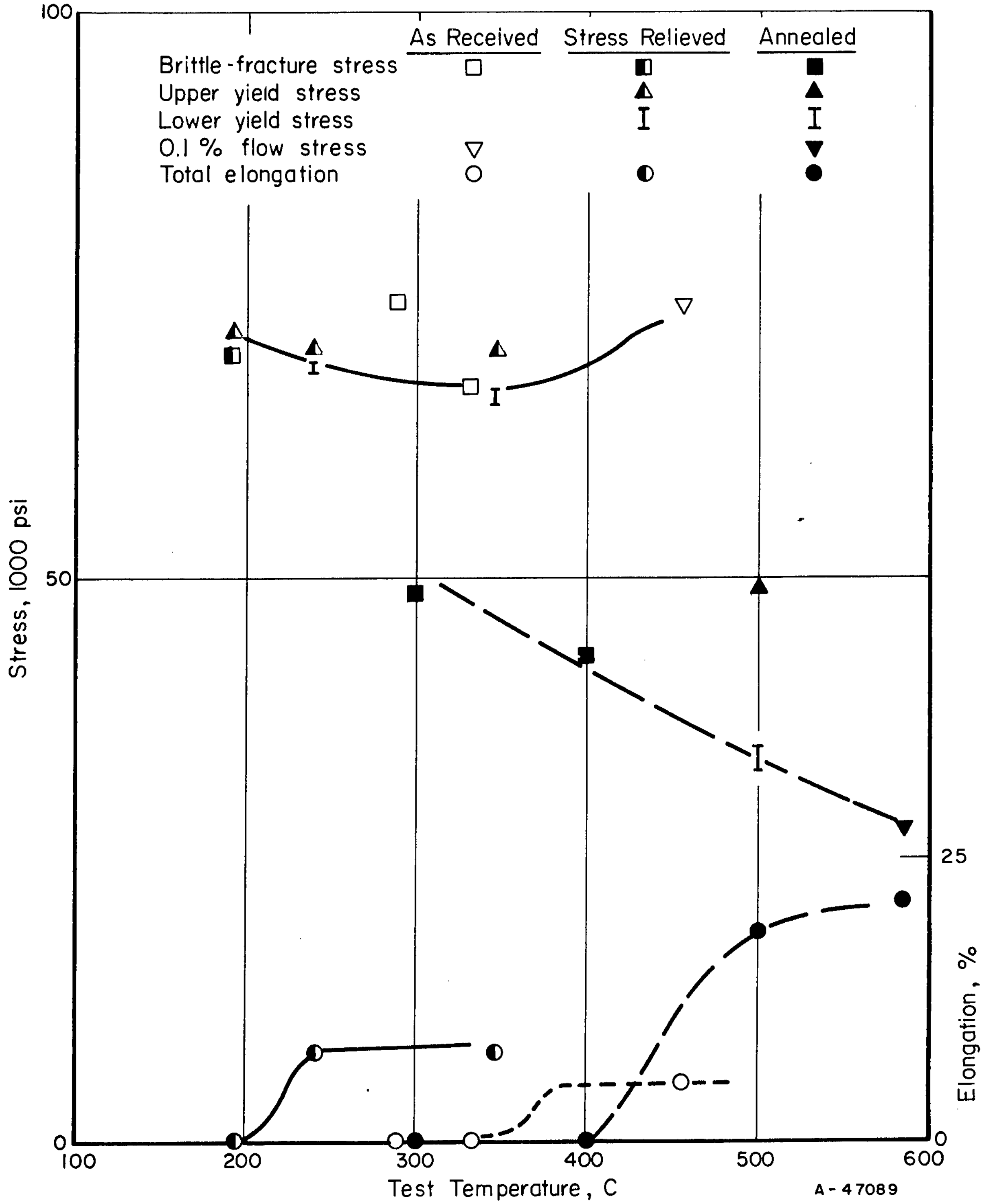


FIGURE 18. MECHANICAL PROPERTIES OF Cr-3 At. % Re AS A FUNCTION OF TEMPERATURE

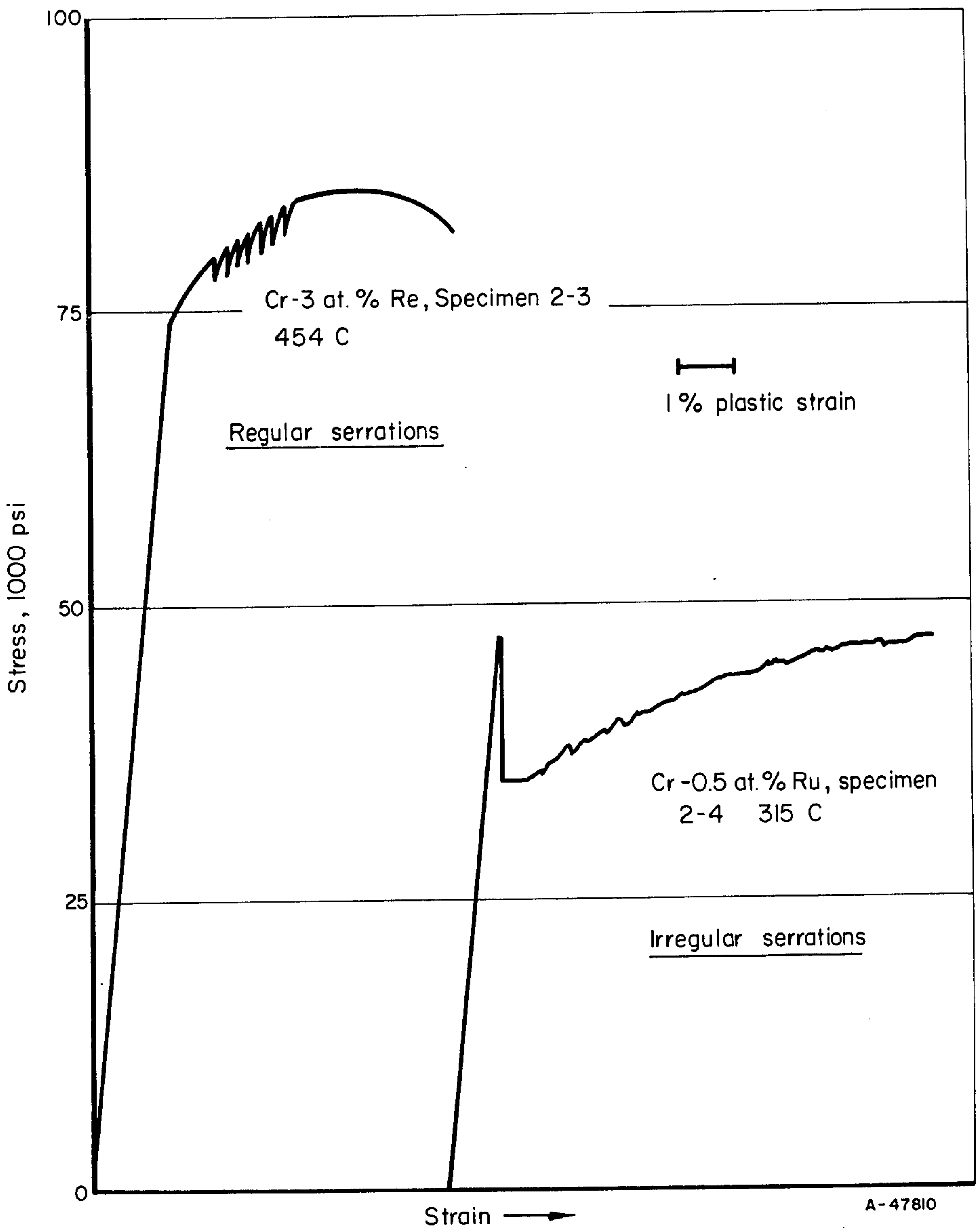


FIGURE 19. EXAMPLES OF REGULAR SERRATIONS AND IRREGULAR SERRATIONS IN THE STRAIN-AGING REGION

DISCUSSION

Table 4 illustrates that the tensile behavior of the as-received versus the recrystallized material is different for all three alloys. The Cr-3 at.% Re behaves in a manner very similar to pure chromium – the as-received material being ductile at a lower temperature than the recrystallized. The Cr-1 at.% Mn recrystallized material has a lower transition temperature than does the as-received, and the Cr-0.5 at.% Ru material has the same transition temperature for the wrought as for the recrystallized material. The reason for this apparently anomalous behavior probably lies in the fact that the as-received material was worked to a different extent in each case. Reference to Figure 13 shows that the Cr-0.5 at.% Ru in the as-received condition is partially recrystallized, the new grains having approximately the same size as those in the annealed material. In Figure 14 it is apparent that the as-received Cr-1.0 at.% Mn has largely recovered to an elongated grain structure, generally smaller than the annealed, whereas Figure 15 shows the as-received Cr-3 at.% Re to be fully worked. As a result of this structure in the as-received condition, the Cr-3 at.% Re alloy showed normal behavior in that the transition temperatures for stress-relieved, as-received, and recrystallized material occurred at progressively higher temperatures.

The transition temperatures of the alloys as a whole show a further grain-size effect. The recrystallized Cr-3 at.% Re has the smallest grain size and the highest transition temperature, while the Cr-1 at.% Mn and the Cr-0.5 at.% Ru have more similar, larger grain sizes and more similar, lower transition temperatures. In addition, the Cr-1 at.% Mn alloy has in places fairly large grains, larger apparently than any present in the Cr-0.5 at.% Ru. Experience with unalloyed chromium[7] has shown that:

(a) Large-grained material is more ductile than fine-grained

(b) Large grains in an otherwise medium-grain sized specimen can lead to somewhat enhanced ductility.

It thus appears that the transition temperatures of the alloys tested in this program reflect to a first approximation the grain size existing in the recrystallized material. One other conclusion which can reasonably be drawn in addition to the effect of grain size is that the Cr-3 at.% Re alloy as tested is intrinsically a more brittle material than the pure chromium. Evidence for this statement is derived from the higher transition temperature of the stress relieved Cr-3 at.% Re (220 C) compared with that of stress-relieved chromium, which in an earlier report was found to be -15 C. This embrittlement seems to be that normally experienced as a result of substitutional alloying but may be exaggerated by the higher oxygen content of this alloy compared to that of the chromium.

It thus appears that hardness-minima alloys have little to offer in the way of enhanced ductility over that of pure chromium. Any effect which does exist probably arises through the effect of alloying on (a) the temperature at which fabrication could be carried out, and thus the amount of work retained in the as-received condition, and (b) the effect of alloying on the recrystallization behavior of the resultant alloy.

The unusual regular serrations observed primarily in the Cr-3Re alloy were similar in nature to those observed in the tensile testing of Cr-35 at.% Re[8] and more recently as reported at higher temperatures by Wilms[11] on dilute chromium alloys.

However, the serrations observed by Wilms occurred at temperatures between 800 and 1000 C as opposed to temperatures of 400 to 600 C in the Cr-35 at.% Re alloys. Although Wilms assumes the effect to be due to strain aging[8], 900 C is a very high temperature for such effects to occur as a result of interstitial interaction, and it seems plausible that such serrated yielding is due to the substitutional atoms. However, at the lower temperatures at which serrations occurred in Cr-35 at.% Re, it seems more reasonable to assume that interstitial atoms are the diffusing species. One final fact which may have a bearing on the mechanism of this serrated yielding is that the regular serrations occur at much higher stresses than do the irregular serrations described both in this report and in Reference 8 on Cr-1Re alloys. It may well be that regular serrations are a phenomenon associated with high stress levels, and irregular serrations with low stress levels. The small number of tests performed, however, do not permit any valid conclusions to be drawn on this issue.

SECTION 3. TENSILE TESTING OF CHROMIUM-17.5 At.% IRON AND CHROMIUM-17.5 At.% RUTHENIUM ALLOYS

INTRODUCTION

It has been appreciated for some time that the brittleness of the Group VI-A refractory metals can be alleviated by additions of 20 to 40 at.% of rhenium. This improvement in ductility, however, can be of only academic interest since rhenium itself is a rare element that is difficult to refine and consequently very expensive. Nonetheless an understanding of the mechanisms of the improvement could lead to development of alloys analogous to the rhenium alloys but utilizing a more common, and therefore cheaper, ductilizing additive.

Within the framework of this argument, the following chromium alloys have been fabricated:

chromium - 35 at.% rhenium*

chromium - 17.5 at.% iron

chromium - 17.5 at.% ruthenium .

The rationale behind the choice of these compositions has been largely in terms of electron to atom (e/a) ratio. Although the electronic structure of the transition metals is incompletely understood, the model of electronic structure typified by the periodic table has been adopted. On this basis the electron-to-atom ratio of the three alloys is ~6.3, iron and ruthenium having twice as many available electrons as rhenium.

Since the mechanism of the rhenium effect is not understood, the current tests were designed to look for similarities or differences in the mechanical behavior of these three alloys in order to provide leads as to the direction of future research.

EXPERIMENTAL PROCEDURE

The Cr-17.5 at.% Fe rod was prepared by a combination of nonconsumable arc melting, hot gas-pressure bonding, rod rolling, and swaging. Arc-melted buttons were crushed, placed in evacuated stainless steel cans, and consolidated at 10,000 psi of helium for 3 hours at 1340 C. The composites were rod rolled to 55% reduction in area at 1200-1300 C and swaged to 70% reduction in area at 1200 C to 1/4-inch-diameter rod.

Cr-17.5 at.% Ru was prepared by nonconsumable melting and casting to a cylindrical shape, canning in stainless steel, rod rolling, and swaging at 1300 C**.

*The mechanical properties of this material were described in the Twelfth and Thirteenth Quarterly Progress Reports.

**A more complete description of these fabrication techniques will be found in a report to Battelle Memorial Institute, written as part of the Integrated Chromium Alloy Program: Third Progress Report, "Preparation, Consolidation, and Fabrication of Cr, Cr-Re, Cr-Mn, and Other Group VI-A Base Alloys".

The Cr-17.5 at.% Fe was tested in tension over the range 300-700 C. The specimen geometry and testing procedure were as described in Section 2 of this report. Since the Cr-17.5 at.% Ru had been successfully fabricated in only small amounts, the procedure used to evaluate this material was to determine the room-temperature ductility in tension as a function of heat treatment.

Isochronal annealing and hardness studies similar to those described in Section 2 showed that the as-received Cr-17.5 at.% Fe rod (which was in a worked condition) recrystallized to an equiaxed structure as a result of a 1-hour anneal at 1300 C. Figure 20 shows the wrought and annealed grain structures of Cr-17.5 at.% Fe and Figure 21 shows the as-received and annealed grain structures of the Cr-17.5 at.% Ru alloy. It is apparent in this latter case that the as-received material had almost completely recrystallized during the high-temperature fabrication.

The results of interstitital analyses for alloy specimens after heat treatment are presented in Table 6.

TABLE 6. RESULTS OF INTERSTITIAL ANALYSIS OF Cr-17.5 At.% Ru AND Cr-17.5 At.% Re ALLOYS

Alloy Composition, at.%		Interstitial Content, ppm				
Nominal	Analysis	C	O	H	N	
Cr-17.5Ru	19.3Ru	~30	87	0.7	20(a)	N.D.(c)
Cr-17.5Fe	17.7Fe	~30	166	0.5	28(a)	90(b)

(a) Vacuum fusion.
(b) Micro-Kjeldahl.
(c) N-D - No Micro-Kjeldahl value was obtained for this alloy due to incomplete solution of the specimen.

EXPERIMENTAL RESULTS

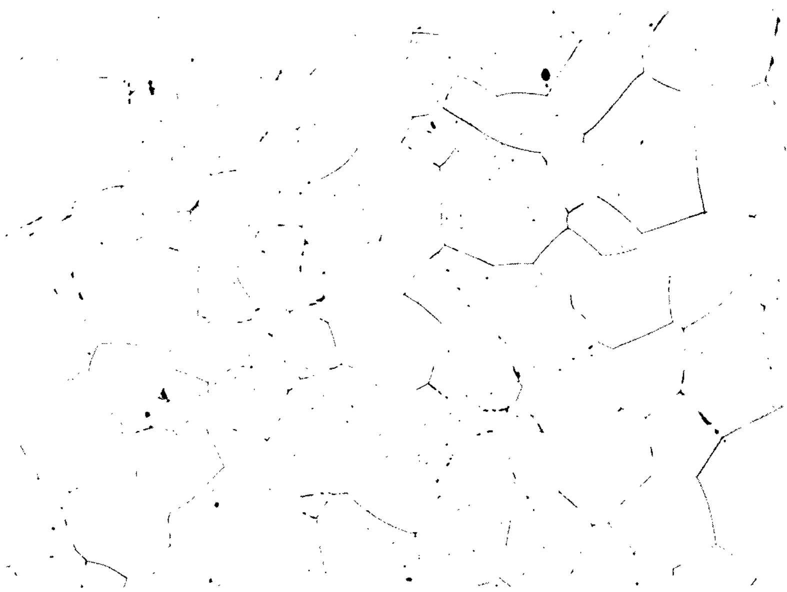
The tensile properties of Cr-17.5 at.% Fe are plotted as a function of temperature in Figure 22 and are tabulated in Table A-3 of the Appendix. The alloy proved extremely strong but was brittle in the recrystallized condition up to a temperature of about 620 C. The wrought material had a ductile-brittle transition at about 540 C, while two specimens tested in the wrought stress-relieved condition indicated that, for material in this heat-treated condition, the transition would be slightly lower again.

The recrystallized material showed a twinning drop as the first sign of plastic deformation over the range 400-600 C and attained a stress of approximately 100,000 psi over this range before twinning occurred. Typical stress-strain curves for recrystallized material are presented in Figure 23, from which it can be seen that after the twinning drop at 400, 500, and 600 C, the stress rose rapidly to a level at or about that of the initial drop before brittle failure occurred. At temperatures above 600 C, the yield stress showed a very strong temperature dependence similar to that shown by unalloyed bcc materials at much lower temperatures, but apparently did not have a similar work-hardening capability since necking occurred immediately after the initial yielding.

This material was unusual in that the wrought specimens exhibited a much greater elongation to fracture than did recrystallized specimens at the same temperature. This can be seen from Figure 22. A wrought sample fractured at 660 C after an elongation of

100X 9177

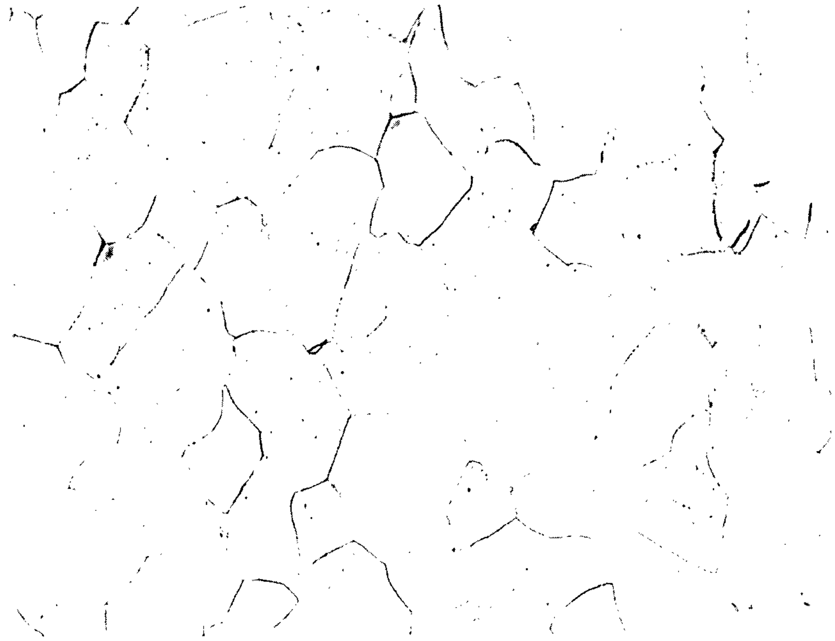
a. Lot 2, As Received



100X 9178

b. Lot 1, Annealed 1 Hour at 1300 C

FIGURE 20. GRAIN STRUCTURE OF Cr-17.5 At.% Fe



100X 9939

a. As Received

100X 9937

b. Annealed 1 Hour at 1400 C

FIGURE 21. GRAIN STRUCTURE OF Cr-17.5 At.% Ru, LOT 2

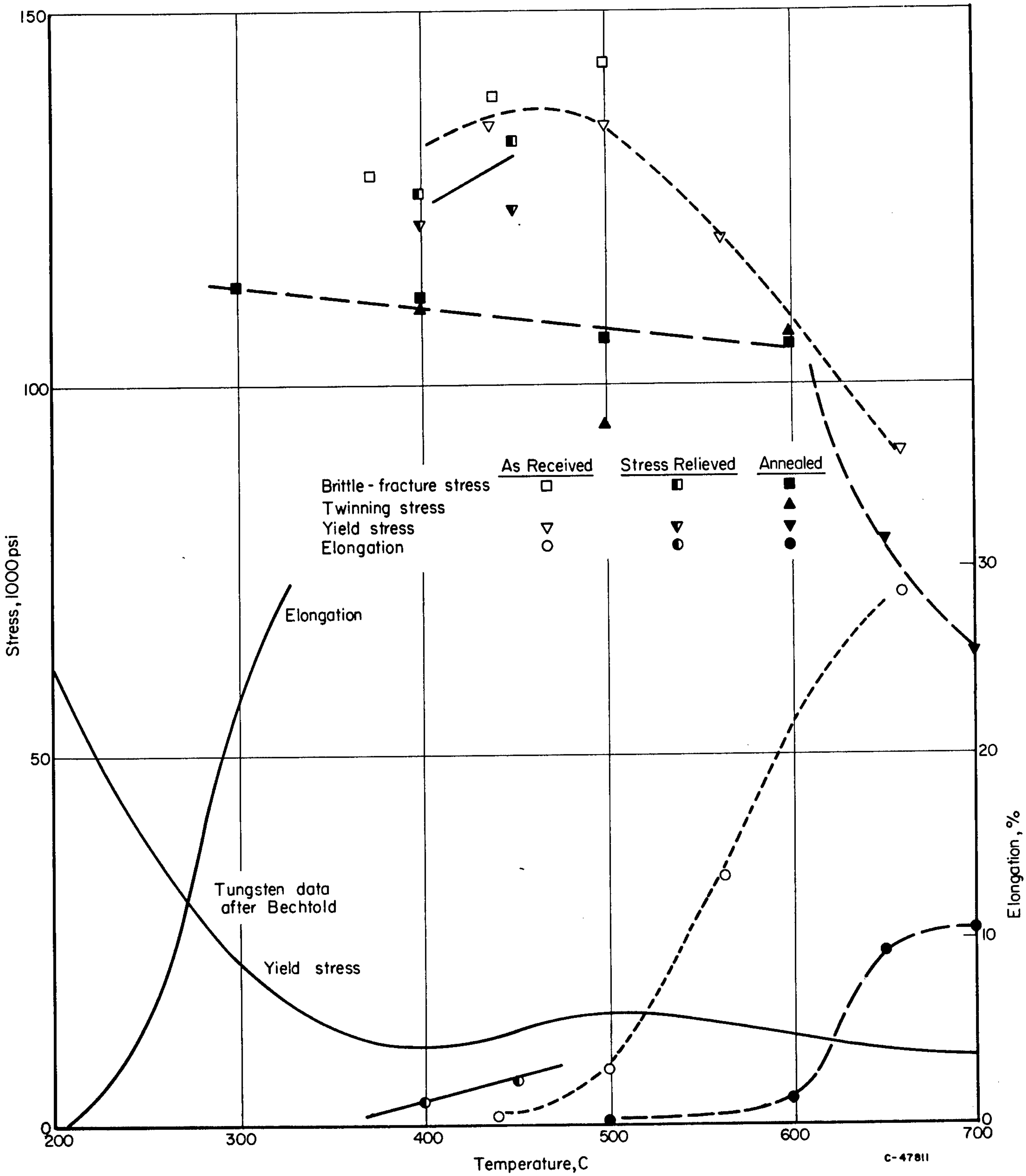


FIGURE 22. TENSILE PROPERTIES OF Cr-17.5 Fe

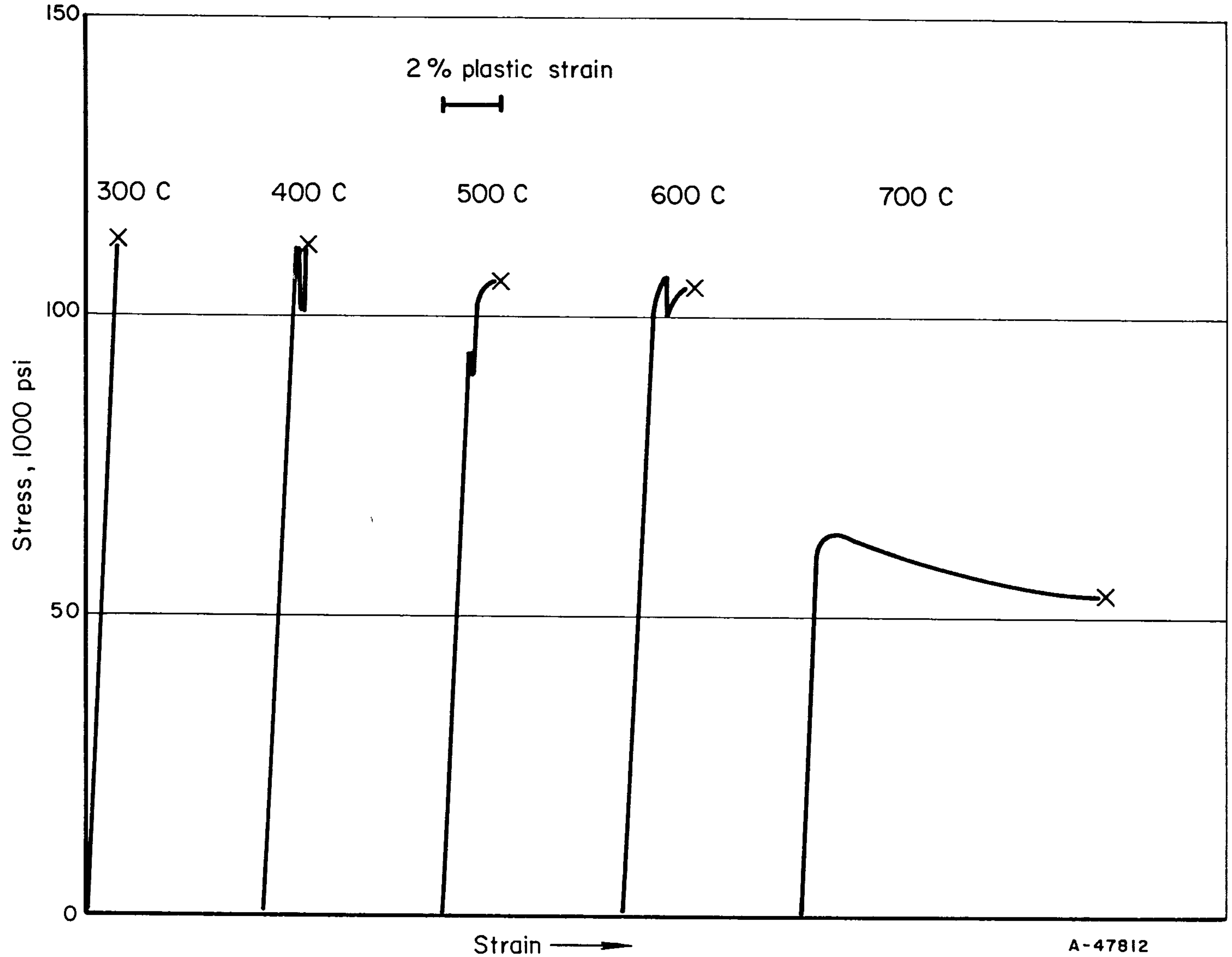
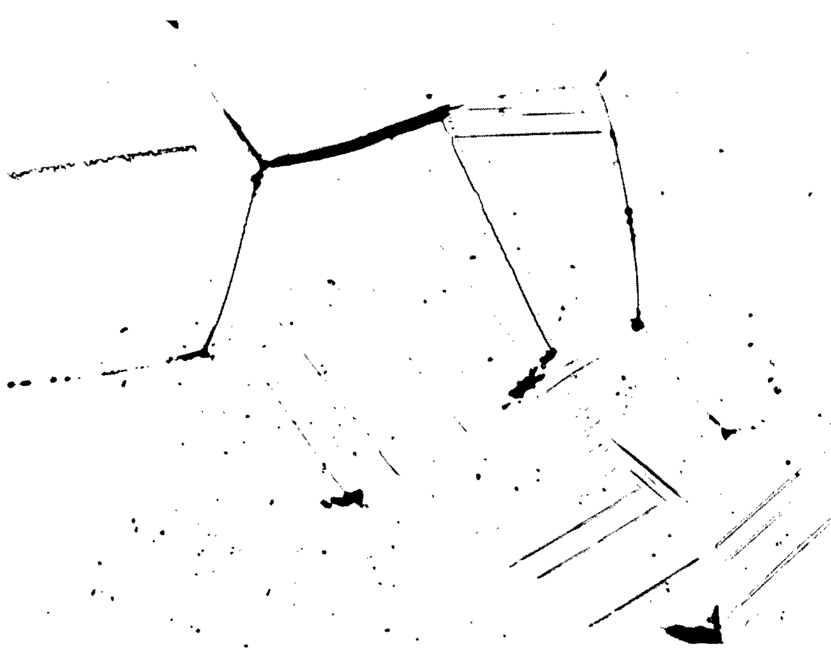


FIGURE 23. TYPICAL STRESS-STRAIN CURVES FOR RECRYSTALLIZED Cr-17.5 At. % Fe

80X 9417

a. Before Etching



400X 9427

b. After Etching

FIGURE 24. METALLOGRAPHIC SECTION THROUGH A RECRYSTALLIZED SAMPLE OF Cr-17.5 At.% Fe TESTED AT 600 C

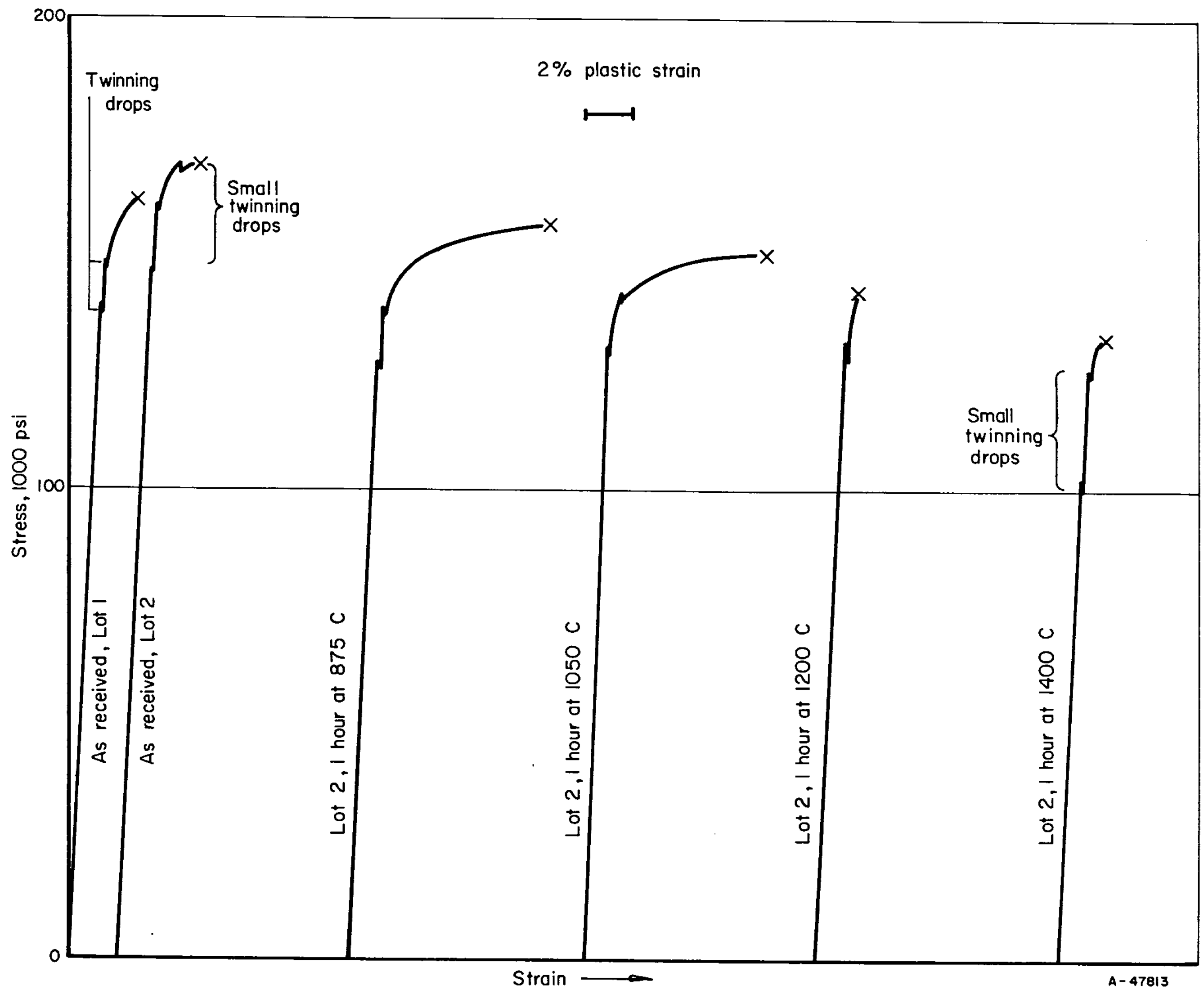


FIGURE 25. EFFECT OF HEAT TREATMENT ON ROOM TEMPERATURE TENSILE PROPERTIES OF Cr-17.5 At.% Ru

29 per cent, whereas a recrystallized sample at 650 C failed after only 9.7 per cent. Metallographic sectioning of recrystallized samples showed that the samples tested between 500 and 700 C had grain-boundary cracks associated with twins. An example of this is shown in Figure 24 for a sample tested at 600 C. The wrought material, however, showed no evidence of cracks prior to etching or evidence of twins after etching.

Figure 25 shows room-temperature stress-strain curves for Cr-17.5 at.% Ru after various annealing treatments. In every case, there is some evidence of ductility, and for stress-relieved material (1 hour at 875 or 1050 C) an elongation of approximately 7 per cent was obtained prior to fracture. On recrystallization, however, the elongation prior to failure was reduced to 0.5 per cent. Qualitatively, in spite of the largely recovered structure in the as-received condition (see Figure 21) the material behaved normally: stress-relieved, as-received, and recrystallized material showed progressively less ductility. Metallographic examination of the specimen fractured after an anneal at 1200 C showed that the gage length contained cracks at twin-grain boundary intersections and also at twin-twin intersections.

DISCUSSION

The tensile behavior of the Cr-17.5 at.% Fe and Cr-17.5 at.% Ru was entirely different, though both alloying additions belong to Group VIII of the Periodic Table. The iron alloy was brittle at temperatures below 500-600 C, whereas the ruthenium had some ductility at room temperature.

In an effort to rationalize this difference, Table 7 summarizes the atomic radii and elastic moduli of chromium, iron, ruthenium and rhenium. It is interesting to note that, with respect to both of these parameters, rhenium and ruthenium, which are beneficial alloying additions, have similar values which are higher than the corresponding values for iron and chromium. Although it would be unwise to speculate on the importance of this fact, it forms a useful framework against which to compare future chromium alloys. It is hoped that if sufficient comparisons of this nature can be made, the material parameters important in ductilizing may emerge.

TABLE 7. CHARACTERISTICS OF CHROMIUM, IRON, RUTHENIUM, AND RHENIUM

Element	Structure	Lattice Spacing, A: c	Lattice Spacing, A: a	Atomic Radius, A	Modulus of Elasticity, 10^6 psi
Cr	Bcc		2.88	1.25	36.0
Fe	Bcc		2.86	1.24	28.5
Ru	Cph	4.28	2.70	1.35	60
Re	Cph	4.46	2.76	1.38	67

The alloys have in common a propensity for twinning, yet the tolerance of the matrix and grain boundaries for twinning is apparently different in the two cases. In the iron alloy, twinning apparently leads to failure through the generation of grain-boundary cracks, while the ruthenium alloy is apparently better able to stand the generation of twins, which leads to useful plastic deformation. The strength levels of the two alloys

are also both similar, although the temperatures of testing were different. In the case of Cr-17.5 at.% Fe, the strength at 600 C was extremely high (~100,000 psi), which substantiates hardness tests on a similar alloy made by Maykuth and Jaffee(12).

In summary, although both alloys show a high strength level and a readiness to twin, the results for the two alloys are different. The ruthenium alloy shows tensile properties which offer good hope of future development to an extent similar to that produced by rhenium, whereas the iron alloy shows a pronounced susceptibility toward grain-boundary failure. It thus appears that the average group number per se is not a basis for production of ductile chromium alloys. Although both ruthenium and iron belong to Group VIII, there may nonetheless exist some basic difference in electronic structure. The magnetic properties of ruthenium and iron, (which are dependent on electronic structure) are also compatible with such a difference, iron being ferromagnetic and ruthenium paramagnetic. If, as still seems likely, electronic structure is important to the deformation mechanism in chromium base alloys, it is through a more subtle mechanism than has as yet been investigated.

SECTION 4. GENERAL DISCUSSION

ALLOYS OF GROUP VI-A METALS WITH RHENIUM

It has been shown that the mechanical properties of chromium, molybdenum, and tungsten are radically changed by adding rhenium in solid solution.

The effect of rhenium on ductility depends markedly on the grain structure and level of interstitial impurity in the base metal. For example, ductility of 99.99+ per cent molybdenum single crystals (expressed as reduction in area) is progressively reduced by alloying with rhenium.[13] However, at all temperatures above 77 K the ductility is good, staying above 40 per cent. On the other hand, the effect of alloying rhenium with 99.9+ per cent purity polycrystalline molybdenum[14] is largely to improve the ductility. Unalloyed molybdenum has some ductility at room temperature but none at 77 K. Alloying with above 10 per cent rhenium progressively improves the ductility at 77 K until at 35 per cent rhenium, it approaches that of the 99.99+ per cent purity alloy. The room-temperature ductility is reduced by alloying with up to about 15 per cent rhenium, but beyond this, the ductility improves up to a maximum at 35 per cent rhenium.

Similarly, the ductility transition temperature of recrystallized chromium is reduced about 300 C by alloying with 35 at.% Re, while in the worked/stress relieved condition this reduction is about 200 C.[8]

The effect of rhenium in improving the low-temperature ductility of Group VI-A metals is also felt in the polycrystalline alloy, W-30 at.% Re, although the lowering of transition temperature is less than in the chromium or molybdenum alloys.[14] By adding rhenium to these metals the ductility transition is made much less precipitous, and instead occurs gradually over wide temperature ranges. Furthermore, the ductility enhancement appears to be maximized at alloy compositions near to the σ-phase boundary.

The work of Pugh[15] demonstrates a beneficial effect of rhenium on ductility in dilute alloys. The room-temperature ductilities of the following materials were compared after annealing at 2850 C:

(1) Commercial tungsten lamp wire ("doped" with potassium, aluminum, and silicon)

(2) Alloy of "doped" tungsten with 3 per cent rhenium

(3) Alloy of "undoped" tungsten with 3 per cent rhenium.

Materials (1) and (3) were fully recrystallized and proved brittle in bending, whereas Alloy (2) which was only partly recrystallized was very ductile. Apparently both the doping elements and 3 per cent rhenium, which mainly retard recrystallization, are necessary for this improvement in ductility. Furthermore, some ductility is retained by Alloy (2) after complete recrystallization.

In addition to this work on dilute W-Re alloys, this report (Section 2) and the previous yearly report have presented the results of tensile tests on dilute Cr-Re and derived alloys. In the chromium-based systems (which were undoped), there is little evidence of any marked improvement in ductility due to alloying at low levels. However, there is again evidence that whatever differences exist rest mainly on the effect of the addition element on the fabrication characteristics and subsequent annealing kinetics of chromium. There is no evidence that such dilute alloys are more resistant to cleavage than is chromium.

One feature of high rhenium alloys is the readiness with which they deform by mechanical twinning. Alloying seems to progressively reduce the average "critical" shear stress for twinning while raising the critical shear stress for slip, at least beyond 5 per cent rhenium. Below 5 per cent rhenium, there is a stress plateau.(13) Room-temperature hardness measurements show qualitatively similar behavior but with the hardness minimum between 0 and 5 per cent rhenium. The consequence is that, at strain rates around 10^{-4} in./in./sec, twins are formed at all temperatures up to 400 C in Cr-35 at.% Re(8), Mo-35 at.% Re(16), and W-30 at.% Re(16). At temperatures in the vicinity of 100 K, the Mo-35 at.% Re and Cr-35 at.% Re alloys actually exhibit slightly lower twinning stresses than do the unalloyed metals. The above effects of rhenium in molybdenum are manifest equally in single crystals(13) and polycrystalline Mo-35 at.% Re(14, 16).

The solubilities of interstitial impurities in molybdenum are below 30 ppm at temperatures below 1200 C(17); therefore, samples of currently available molybdenum are usually saturated and contain precipitates. Klopp et al.(16) reported that the oxide precipitate MoO_2 tends to accumulate at grain boundaries as a continuous film, and that alloying with more than 10 per cent rhenium aggregates the oxide into globules which have a large angle of contact with the grain boundary. These globules are dispersed both in the matrix and at the grain boundaries. Klopp concluded that the chemical composition of the globules was approximately $MoReO_4$. Thus, alloying changes the chemistry and morphology of the grain-boundary oxides in molybdenum. In 99.9 per cent purity molybdenum stressed below about 150 K, and in 99.99 per cent purity chromium stressed below 300 K, grain-boundary rupture precedes gross deformation and the material is brittle. However, in high-rhenium alloys, although grain-boundary rupture does occur, it does so at higher stresses and is preceded by gross plastic deformation.(8, 16) Alloying appears to inhibit cleavage because fractures of the alloys are generally complete grain-boundary failures. The only example of cleavage observed so far in high-rhenium alloys is described in this report. The failures examined previously have consisted entirely of grain-boundary facets. While alloying is beneficial in redistributing oxides, it is reportedly not effective with carbides and nitrides.(16)

Evidence from the electron microprobe indicates a higher than average concentration of rhenium at grain boundaries.(16) However, it is not clear whether this is an effect separate from the high concentration of rhenium in the oxides present at the grain boundaries. This grain-boundary effect has not been confirmed by recent experiments on chromium-rhenium.(18) Features have been observed at grain boundaries by field-ion microscopy(19) which may indicate some segregation of rhenium within a few atomic distances of a boundary. However, it must be emphasized that the interpretation is not rigorous. No X-ray evidence of long-range order was detected in the Mo-35 at.% Re alloy. The degree of short-range order present is not known. The conclusion of Klopp et al.(16) that there is an absence of short-range order is invalid since the experiment used was incapable of detecting it. In fact, some degree of short-range order has generally been detected in all alloy systems that have been examined for atomic ordering.

The most valuable practical aspect of the rhenium-alloying effect is the improvement in low-temperature ductility in Group VI-A metals of moderate purity. It would probably be useful to critically review the hypotheses that were advanced in 1958 to account for this.

Hypothesis 1: Alloying With Rhenium Causes a Change in the Precipitate Morphology Such That it Promotes Ductility

Certainly, the currently available Group VI-A metals have notoriously weak grain boundaries, from which sites brittle failures appear to originate. The report of Klopp et al.[16] describes how a continuous film of oxide at the grain boundaries in molybdenum is replaced by a globular phase in Mo-35 at.% Re. The suggestion that this should enhance the strength of the grain boundary is very plausible. However, this cannot be the only factor, since while cleavage is the normal mode of crack propagation in molybdenum it has not as yet been observed in Mo-35 at.% Re. Clearly, alloying has developed a strong resistance to cleavage, which must also contribute to ductility.

The hypothesis went on to suggest that the agglomeration of precipitate arises from an increase in the angle of contact, which in turn arises from a reduced grain-boundary energy. Allen[20] at Battelle has obtained some preliminary experimental evidence that the grain-boundary energy is measurably reduced vis-a-vis the surface energy. This point is under intensive study at present.

However, the above observations have not been corroborated by other workers. No evidence has been found of a continuous impurity film in the grain boundaries of chromium, nor is it established that the precipitates are agglomerated to a greater extent in Cr-35 at.% Re than in unalloyed chromium. The impurity phases in these materials are now under investigation as another phase of the Integrated Chromium Alloy Program, but meanwhile this hypothesis must be regarded as unproven.

Hypothesis 2: Alloying With Rhenium Improves Ductility by Lowering the Solubility of Interstitial Impurities

to p45

This hypothesis is actually twofold: that the solubility is lowered, and that the result of a reduced solubility is an increase in ductility.

This hypothesis appears to have had its origin in the Cottrell theory of the yield point and brittle fracture. The crux of this theory was that the phenomena of the yield point and brittle fracture were entirely due to the immobilization of dislocations by impurity atmospheres that had formed at the expense of soluble interstitial atoms. The argument ran that if the amount of interstitial in solution were reduced, the extent of dislocation locking would be likewise reduced leading to a diminution of the tendency for discontinuous yielding and brittle fracture. However, the current theory of yielding and crack propagation considers that the pinning of dislocations is not the only factor which determines the yield point and the tendency towards brittle fracture, so the original basis for this hypothesis is now accepted less widely. In fact, an argument can be made that good ductility is exhibited by bcc metals only when they exist as a single bcc phase and that when impurity phases are present as well, the ductility deteriorates. With the

current state of industrial practices, the commercially produced metals are single phase only when their interstitial solubilities are high (e.g., vanadium, columbium, and tantalum), and significantly their ductility also is high. On the other hand, the metals with low solubilities are generally available only as multiphase materials due to the presence of precipitated impurities, and accordingly these metals (chromium, molybdenum, and tungsten) have high transition temperatures, of the order of 300 K. Iron is an intermediate case. Furthermore, Lawley et al.[21] have shown that if polycrystalline molybdenum is purified past the limits of chemical analysis it has ductility down to below 4 K. Therefore, at this time it would seem to be more likely that good ductility is associated with increased rather than reduced solubility. It should be noted, however, that the more ductile Cr-35 at.% Re does contain impurity phases.

There is no known experimental evidence to indicate that the already low solubilities of chromium, molybdenum, and tungsten[17] are further reduced by alloying with rhenium. On the other hand, there are three pieces of evidence to indicate that the solubility of interstitial impurities is actually increased:

(1) Experiments on Snoek damping due to nitrogen in chromium and Cr-35 at.% Re have been carried out at Battelle by Klein and Clauer.[22] The interpretation of the results, although tentative at present, indicates that after furnace cooling from typical recrystallization temperatures, the amount of nitrogen retained in solution in Cr-35 at.% Re is about 1000 times greater than in unalloyed chromium.

(2) The height of the strain-aging peaks (on the graph of tensile flow stress versus temperature) are considerably increased by adding 35 at.% rhenium to chromium[8] and molybdenum[23]. The phenomena of dynamic strain aging, according to the current theories of both Cottrell[24] and Schoeck and Seeger[25], is due to interstitial impurities in solution, and, other things being equal, the increase in strength is proportional to the interstitial-solute concentration. Accordingly then, these observations suggest that the concentration of retained interstitial solute is higher in the high-rhenium alloys than in the respective pure metals.

(3) Bryant[26] has investigated the solubility of oxygen in alloys of the bcc crystal structure extending from an average group number, N, of 5.0 to 6.2. Similarly, Jones[27] has measured the hydrogen solubility in bcc alloys from an average group number of 5.0 to 6.4, and has correlated this with the height of the density of states curve $n(E_f)$, at the Fermi level, as inferred by a variety of physical-property measurements, such as magnetic susceptibility, low-temperature specific heat, and the superconducting transition temperature; the correlation is good. The solubilities and $n(E_f)$ have large values at $N = 5.0$, above which they fall progressively until at about $N = 5.7$ they reach a minimum which persists until about $N = 6.0$. Then an increase occurs which leads to a small maximum at $N = 6.25$. The hydrogen-solubility measurements between $N = 6.0$ and $N = 6.4$ were actually carried out on Mo-Re alloys and so are particularly relevant to this discussion. If it is accepted that the variation of $n(E_f)$ may be correlated with the solubility of all interstitial elements, in addition to the specific cases treated by Bryant and Jones, then one is forced to conclude that the interstitial solubilities in the high-rhenium alloys (with N between 6.25 and 6.4) are greater than in the corresponding Group VI-A metal.

In order to settle this solubility question, further experiments on Snoek damping are in progress by Klein, and in addition, plans are afoot for some direct solubility measurements on chromium and Cr-35 at.% Re.

Hypothesis 3: The Ready Occurrence of Deformation Twinning in High-Rhenium Alloys Promotes Ductility

It was postulated that the appearance of twinning as an additional deformation mode promoted ductility in the alloys by initiating plastic flow at stresses low compared to those for slip, thereby offsetting to some extent the effect of solid-solution hardening. Certainly, this mechanism would increase the strain to fracture: necking and subsequent ductile fracture occurs at the point where the rate of strain hardening, $\frac{d\sigma_f}{d\epsilon}$, is equal to the flow stress σ_f.[28] Now, in Mo-35 at.% Re at 300 K, the contribution of twinning to the strain is a maximum at small strains and thereafter diminishes gradually, providing a negligible contribution at about 10 per cent.[29] Since twinning occurs at slightly lower stresses in the rhenium alloys, compared with the pure metals, whereas slip takes place at higher stresses (due to solid-solution hardening), then the high-rhenium alloys should exhibit high rates of work hardening as twinning gradually gives way to slip. Consequently, the onset of necking should be postponed until the strain-hardening rate has diminished to the point where $\frac{d\sigma_f}{d\epsilon} = \sigma_f$. If ductility is defined in terms of strain to fracture, then twinning does improve ductility.

However, twinning per se does not always do this, because although the above argument is quite general, it will not apply if the appearance of the first twin induces brittle fracture. There is no one-to-one relation between prolific twinning and ductility. For example although in the high-rhenium alloys there does appear to be such a correlation, in the alloys Cr-35 at.% Mn[30], Cr-35 at.% Fe[12, 30], and Cr-17 at.% Fe[31], prolific twinning appears to generate many microcracks at temperatures up to 600 C. Accordingly, then, there must be some more basic property of the high-rhenium alloys that allows them to withstand the very rapid loading rates that are involved locally in twin formation.

Reid[30] has recently examined the dislocation arrangements that are produced by the room-temperature deformation of Cr, Cr-35 at.% Re, and Cr-35 at.% Fe. The dislocations in the alloys are arranged very differently from those in unalloyed chromium; in the pure metal they are arranged in a cellular pattern, are very tangled, and appear to move at low stresses. On the other hand, in the alloys dislocations are distributed uniformly, are predominantly in the screw orientation, and are moved with difficulty. The dislocation arrangements of Cr-35 at.% Re and Cr-35 at.% Fe after the same deformation are rather similar, but the ductilities are very dissimilar. It is concluded that the dislocations in these alloys are characteristic of easy twinning rather than high ductility.

The observations made with the electron microscope were unable to establish the types of slip plane that are preferred in the rhenium alloys, due to the absence of discrete slip bands in the condition that was studied. However, the twinning planes were shown to be consistent with $\{112\}$-type planes.

The above observations were essentially static, but the crux of the ductility question may be the dynamic behavior of dislocations, since deformation, crack nucleation, and propagation all involve moving dislocations. Some theoretical work by Hahn et al.[32] has suggested that the most important dynamic parameter is m in the equation

$$\bar{v} = \left(\frac{\sigma}{\sigma_o}\right)^m ,$$

where $\bar{v}$ is the average dislocation velocity under a stress σ (σ_o is just a constant of the material). Briefly, it is argued that large values of m favor ductility (or more specifically resistance to crack propagation), and that the value of m may be inferred from the relation between yield stress and strain rate. Measurements of this relation by Reid[33] on chromium suggest that the corresponding value of m is about 7, whereas for Cr-35 at.% Re Gilbert[8] concludes that $m \approx 100$. These values correlate well with the brittleness of chromium and the ductility of Cr-35 at.% Re, and in particular with the reluctance of high-rhenium alloys to undergo cleavage. However, before this is taken too seriously, it is intended to see if this correlation between m and ductility is general and applies to other alloys as well. Also, further experimental data are needed to clarify the importance of σ_o. It is though that this has some relevance too, because highly purified niobium[34] and iron[35] have small values of m (~6), which can not be equated with brittleness; on the other hand, purification reduces the value of σ_o, and this may mitigate the effect of a small m. However, this correlation is highly tentative at present, and work is in progress to test it further.

It has been suggested by Jaffee et al.[14] that the preponderance of twinning in the high-rhenium alloys is due to lowering of the twin-matrix interface energy, and the energy of the stacking fault created by the movement of a partial twinning dislocation. However both Rosenfield[36] and Reid[30] have observed hexagonal networks in high Cr-Re alloys and have not seen any visible extension of the triple nodes, and Ogawa and Maddin[37] and Votava[38] have had similar experience with Mo-35 at.% Re. This puts a lower limit of about 50 ergs/cm^2 on the stacking-fault energy but does not establish whether or not a reduction has taken place. Ralph and Brandon[19] interpret their field-ion-microscope images of deformed W-26 at.% Re in terms of closely spaced stacking faults, but on the other hand X-ray analysis of a broken Cr-35 at.% Re specimen failed to reveal any line shifts or peak broadening consistent with the presence of stacking faults.[39] Allen at Battelle is currently engaged in thermal-grooving experiments on twin boundaries with a view to measuring their energy. However, at this time it is not established whether rhenium lowers the stacking-fault energy, and the only thing known about this energy is that it is greater than about 50 ergs/cm^2.

Thus it is possible only to reach tentative conclusions at present regarding the hypothesized ductilizing factors of rhenium alloying. Hypothesis 1 is still tenable, although no further supporting evidence has been found following that reported by Klopp et al.[16]. Hypothesis 2 appears to be wrong, and in fact the interstitial concentration retained in solution is probably increased. The ready occurrence of twinning (Hypothesis 3) cannot of itself be a ductilizing factor. There must be some even more basic change in deformation behavior that allows the lattice to withstand twin formation without cracking. It is suggested that this factor may involve the parameters m and σ_o, as defined above.

End

Current and Future Work

In addition to the hypotheses already discussed, it is also possible to pass preliminary judgement on the usefulness of electron-to-atom (e/a) ratio as a formula for producing ductile alloys. The results presented in Section 3 of this report show that alloying with iron and ruthenium, both from Group VIII of the periodic table, produces vastly different results. The implication of this is that a certain e/a per se cannot lead to a useful ductilizing recipe, but it may well be that the general idea of increasing e/a beyond the density of states minimum at e/a = 6 could lead to increased solubility (as outlined in the discussion of Hypothesis 2) which, in turn, could enhance ductility. This increase in e/a can take place only up to the alloying addition at which the α phase becomes less stable than the σ. In other words, a promising range of alloys to be investigated could be those near to the α/σ phase boundary. According to this rationale, the Cr-Ru system at rhenium additions closer to the phase boundary seems to be most promising in view of the already tolerable room-temperature ductility of the Cr-17.5 at.% Ru alloy. Such alloys will be screened during the current year's work.

Other lines of investigation which are being pursued or which will be pursued during the course of the year's work are the effect of rhenium on the active slip planes in Mo-35 at.% Re and the effect of rhenium on the cleavage plane — now that it has been shown, in Section 1, that cleavage can occur in rhenium alloys. Steps are also being taken to grow single crystals of Cr-35 at.% Re by an induction-heating process in a purified hydrogen atmosphere, in an effort to repeat this work on chromium-base material. Knowledge pertinent to these mechanical properties of the rhenium alloys should provide a basis for replacing hypotheses which have been disproved or on which doubt has been cast.

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APPENDIX

TABLE A-1. NOTCH-IMPACT PROPERTIES OF CHROMIUM AND Cr-35 At.% Re

Lot	Sample	Test Temp, C	Absorbed Energy, inch-lb	Classification of Sample
Cr-1	P97	221	10	2 pieces, brittle
1	P101	227	60	2 pieces, semiductile
1	P100	260	>169	65° bend, ductile
1	P98	316	74	2 pieces, semiductile
1	P99	349	>200	65° bend, 1 piece
1	P96	404	>187	65° bend, 1 piece
Cr-2	P83	216	1	2 pieces, brittle
2	P29	254	2	2 pieces, brittle
2	P24	266	1	2 pieces, brittle
2	P86	271	2	2 pieces, brittle
2	P32	277	78	2 pieces, semiductile
2	P31	288	>154	70° bend, ductile
2	P88	288	116	2 pieces, semiductile
2	P84	293	>190	70° bend, ductile
2	P81	304	4	2 pieces, brittle
2	P27	304	4	2 pieces, brittle
2	P24	316	>185	70° bend, ductile
2	P87	332	8	2 pieces, brittle
2	P23	338	100	2 pieces, semiductile
2	P25	360	7159	70° bend, ductile
2	P28	366	>152	70° bend, ductile
2	P85	371	>66	75° bend, ductile
2	P82	410	>100	65° bend, ductile
Cr-3	P89	340	11	2 pieces, brittle
3	P93	343	14	2 pieces, brittle
3	P94	375	16	2 pieces, brittle
3	P95	388	>198	70° bend, ductile
3	P33	410	>182	70° bend, ductile
3	P90	415	>52	80° bend, ductile
3	P92	418	>198	70° bend, ductile
3	P91	427	>154	70° bend, ductile
Cr-35Re-1	Imp 1	24	4	2 pieces, brittle
	Imp 3	207	9	2 pieces, brittle
	Imp 5	312	18	2 pieces, brittle
Cr-35Re-2	Imp 2	164	5	2 pieces, brittle
	Imp 4	316	>152	1 piece, semiductile[(a)]
	Imp 6	395	195	2 pieces, ductile

(a) This sample was broken by hand at room temperature into 2 pieces.

TABLE A-2. HEAT TREATMENT AND TENSILE

Specimen Composition	Fabrication History	Lot	Heat Treatment	Specimen No.	Cross Sectional Area of Gage, sq in.	Test Temp, C	Twinning	Brittle Fracture
Cr-0.5 at.% Ru	Swaged 1200 C	1	Annealed 1300 C	0.5 Ru 1-1	.00622	400	--	--
				1-2	.00660	300	--	33.3
				1-5	.00641	153	--	30.5
				1-6	.00636	500	--	--
				1-8	.00642	590	--	--
				1-9	.00652	241	--	--
				1-11	.00643	350	--	--
	Swaged 900 C	2	As received	0.5 Ru 2-4	.00637	315	--	--
				2-5	.00700	168	--	34.3
				2-7	.00740	246	--	--
Cr-1 at.% Mn	Swaged 1200 C		As received	1 Mn 1	.00679	205	--	60.0
				10	.00690	101	--	68.1
			Stress relieved 875 C	1 Mn 3	.00595	Room	--	59.0
				4	.00709	193	--	59.2
				5	.00684	400	--	62.2
				6	.00692	212	--	62.0
				7	.00690	245	--	--
				8	.00688	297	--	--
				9	.00680	85	--	55.1
			Annealed 1300 C	1 Mn 11	.00700	102	--	--
				13	.00681	250	--	--
				14	.00668	Room	--	48.0
				15	.00708	68	--	55.5
				16	.00685	143	--	--
Cr-3 at.% Re	Swaged 1200 C	1	Annealed 1300 C	3 Re 1-1	.00423	400	--	43.3
				1-2	.00608	500	--	--
				1-3	.00571	584	--	--
				1-6	.00660	300	--	49.0
	Canned in stainless steel	2	As received	3 Re 2-1	.00712	290	--	74.5
				2-2	.00700	330	--	67.0
				2-3	.00707	454	--	--
		2	Stress relieved 875 C	3 Re 2-5	.00696	239	--	--
				2-6	.00703	194	--	69.7
				2-7	.00762	347	--	--
	Canned in molybdenum	3						Very poor

PROPERTIES OF DILUTE CHROMIUM-BASE ALLOYS

Stress, 1000 psi								
0.1% Flow	Upper Yield	Lower Yield	3% Flow	5% Flow	8% Flow	Ultimate Tensile	Uniform Elongation, %	Total Elongation, %
--	--	17.7-20.0	30.3	32.2	33.8	36.8	20.0	35.0
--	--	--	--	--	--	--	0	0
--	--	--	--	--	--	--	0	0
--	21.6	19.7-20.8	36.2	38.4	39.9	40.8	15.0	35.0
21.5	--	--	31.2	31.3	31.5	31.5	10.0	47.0
--	--	18.7-20.7	28.4	31.4	33.8	38.5	27.0	41.0
--	22.9	19.5-21.3	32.2-33.4	34.0	35.4	38.2	22.0	35.0
--	47.1	35.0	41.7	45.5	48.2	52.0	21.5	35.3
--	--	--	--	--	--	--	--	--
--	34.1	31.4-32.8	38.1	41.2	43.3	47.3	--	52.5
47.2	--	--	59.2	--	--	--	3.5	3.5
--	65.5	64.7	--	--	--	--	1.0	1.0
--	59.5	59.0	--	--	--	--	--	0.25
--	--	49.4-50.8	57.9	--	--	--	4.0	4.0
44.8	--	--	60.3	62.0	--	--	5.25	5.25
--	50.6	49.7	58.7	61.1	--	--	6.5	6.5
--	42.1	40.7	50.1	52.6	53.6	53.6	9.0	21.0
--	47.7	47.1	57.6	60.1	59.6	60.4	6.3	18.0
54.6	--	--	--	--	--	--	0.25	0.25
--	--	38.7-41.2	48.5	51.9	54.3	55.8	17.6	32.2
--	37.5	33.4	43.9	46.5	48.3	48.9	12.0	24.2
--	42.1	39.3-40.0	--	--	--	--	--	2.0
--	44.1	38.2	49.5	53.8	--	--	6.2	6.2
--	--	34.2-35.1	46.0	49.0	51.3	52.6	18.7	36.1
--	--	--	--	--	--	--	0	0
--	49.2	32.9-37.8	51.0	55.1	56.7	56.7	8.6	18.5
28.1	--	--	57.0	59.4	59.0	59.4	5.5	21.5
--	--	--	--	--	--	--	--	--
--	--	--	--	--	--	--	0	0
--	--	--	--	--	--	--	0	0
74.3	--	--	83.7	81.5	--	84.2	2.5	5.0
--	70.4	68.4-69.3	75.4	78.0	78.3	78.8	7.0	8.0
--	71.9	--	--	--	--	--	0	0
--	70.2	66.0-67.1	72.4	72.9	68.8	73.6	4.0	8.0

specimens, cracked all the way through - none tested

TABLE A-3. HEAT TREATMENT AND MECHANICAL

Specimen Composition	Fabrication History	Lot	Heat Treatment	Specimen No.	Cross Sectional Area of Gage, sq in.	Test Temp, C	Twinning
Cr-17.5 at.% Ru	Swaged 1300 C	1	As received	17.5 1	.00707	Room	138.5
	Swaged 1300 C	2	As received	17.5 1	.00714	Room	147.0
			875 C stress relieved	2	.00682	Room	127.0
			1050 C anneal	3	.00670	Room	129.7
			1200 C anneal	4	.00690	Room	131.0
			1400 C anneal	6	.00674	Room	102.0
Cr-17.5 at.% Fe	Swaged 1200 C		1300 C	17.5Fe 1-3	.00622	400	110.5
				1-4	.00619	300	--
				1-5	.00660	650	--
				1-7	.00641	500	94.4
				1-8	.00635	600	107.0
				1-11	.00627	700	--
	Swaged 900 C	2	As received	17.5Fe 1	.00650	373	--
				2	.00657	440	--
				3	.00657	563	--
				4	.00650	660	--
				6	.00650	505	--
		2	Stress relieved 875 C	17.5Fe 7	.00738	404	--
				8	.00712	449	--

PROPERTIES OF Cr-17.5 At.% Ru AND Cr-17.5 At.% Fe

Stress, 1000 psi									
Brittle Fracture	0.1% Flow	Upper Yield	Lower Yield	3% Flow	5% Flow	8% Flow	Ultimate Tensile	Uniform Elongation, %	Total Elongation, %
162.0	--	--	--	--	--	--	--	1.5	1.5
169.5	--	--	--	--	--	--	--	1.3	1.3
156.0	--	--	--	151.5	154.5	--	--	7.0	7.0
151.0	--	--	--	149.0	150.0	--	--	7.0	7.0
142.0	--	--	--	--	--	--	--	0.5	0.5
132.0	--	--	--	--	--	--	--	0.5	0.5
111.2	--	--	--	--	--	--	--	--	0.3
113.0	--	--	--	--	--	--	--	0	0
--	--	79.5	--	74.0	72.7	70.0	--	--	9.7
106.0	--	--	--	--	--	--	--	--	0.4
105.0	--	--	--	--	--	--	--	--	1.0
--	--	64	--	61.0	60.6	58.5	--	--	10.5
128.0	--	--	--	--	--	--	--	0	0
138.0	--	--	135.0	--	--	--	--	0.5	0.5
--	106.0	--	--	118.0	116.5	111.0	120.0	0.5	13
--	86.0	--	--	93.0	92.0	89.0	96.0	2.0	29.0
143.0	134.0	--	--	143.0	--	--	--	3.0	3.0
125.8	--	--	121.9	--	--	--	--	1.4	1.4
132.9	--	--	123.6	--	--	--	--	2.5	2.5

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